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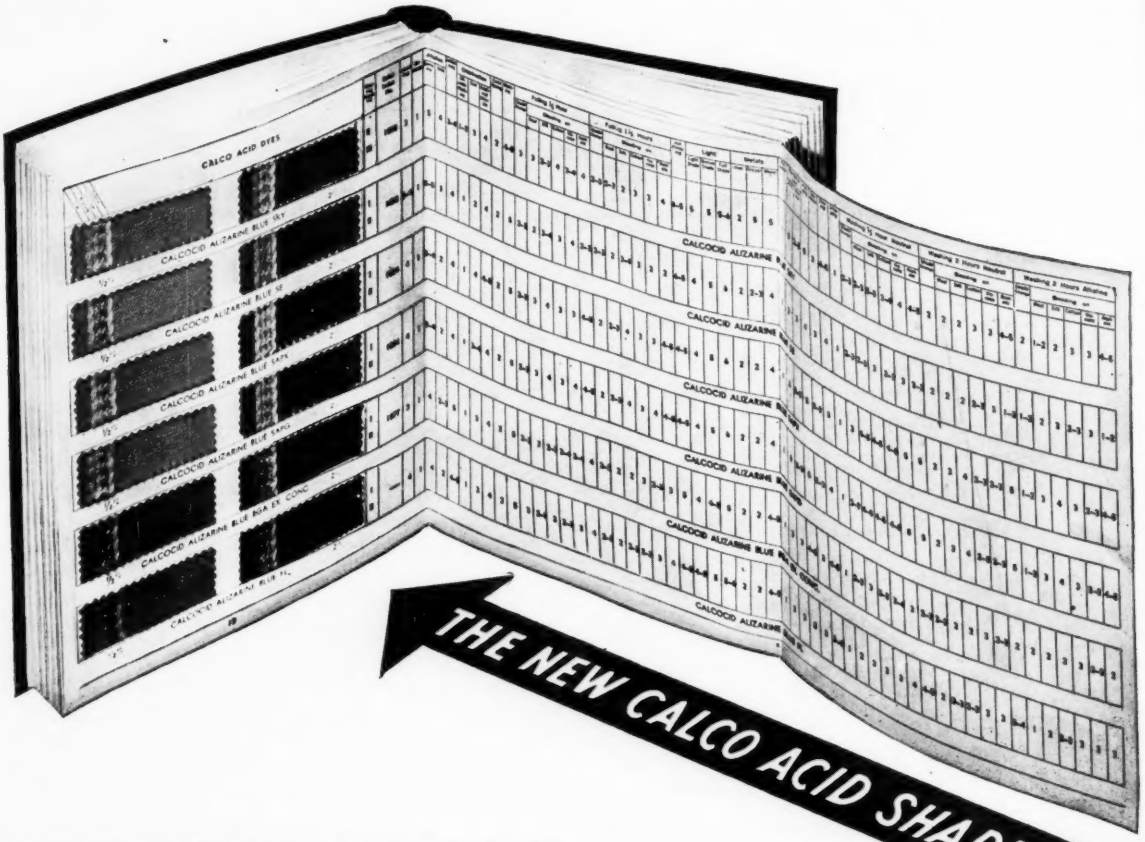
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VOLUME 29 JUNE 10, 1940 NUMBER 12

Microscopic Examination of

Cotton Fibers

in Cuprammonium Hydroxide Solutions

CHARLES W. HOCK and MILTON HARRIS*

ABSTRACT

It has been known for nearly a century that cuprammonium hydroxide solutions have a pronounced effect upon the cell walls of plants. Many investigators have observed, for example, that when cotton fibers are treated with this reagent, the bulk of the fiber appears to be dissolved, thereby producing a viscous solution from which cellulose can be precipitated by various means. The solubility of cellulose under these conditions has been utilized industrially in the manufacture of artificial textile fibers, and also in the laboratory as a means of evaluating the quality of the fiber, especially as regards tensile strength.

There are two different points of view concerning the behavior of the cellulose fibers when treated with cuprammonium hydroxide solutions. According to one group of investigators the cellulose of the cotton fibers does not dissolve in the reagent but maintains a visible state of aggregation in the form of diminutive particles, each of which is surrounded by cementing material which swells in cuprammonium solutions, thereby giving rise to a gel-like structure upon which many of the characteristic properties of the dispersion depend. The other group of investigators has supported the view that cellulose is dissolved in cuprammonium hydroxide, and that properties of the solutions, such as viscosity, are functions of the chain-length of the cellulose molecule.

Since the above viewpoints lead to widely different interpretations of the behavior of cotton in this solution, it appeared advisable to undertake a new investigation of the microscopically observable changes which cotton fibers undergo in this reagent.

It was found that during the treatment of cotton with cuprammonium hydroxide solutions, the cellulose dissolves leaving residues which vary in amount and in structure depending upon the degree of purification of the fibers. The undissolved residue from raw and from dewaxed fibers consists principally of fragmented shells which formed the outer surface of the fibers, and to a lesser extent of material from the lumen. Both of these residues are isotropic and stain deeply with ruthenium red. Fibers from which both wax and pectic substance have been removed dissolve in amount of isotropic residue which exhibits no definite cuprammonium reagent, leaving only a very small cytological structure.

When a steady flow of cuprammonium hydroxide solution is applied to fibers mounted on a slide, the initial swelling of the fibers is followed by the appearance of small ellipsoidal particles which range from 1-2 μ in size, appear bright between crossed nicols, and become dark in color when stained with iodine and sulfuric acid. Identical particles appear, however, when cuprammonium hydroxide solution is drawn under the coverglass, in the absence of fibers. These particles, which result from the exposure of the cuprammonium hydroxide reagent to the air, are then washed under the coverglass where they become intimately associated with the fiber residue. Formation of these particles may be prevented by using a specially constructed cell which prevents exposure of the reagent. Under these conditions, no particles were observed during the slow passage, even for prolonged periods, of the cuprammonium reagent over the cotton fibers.

I. INTRODUCTION

THE viscosity of cuprammonium hydroxide solutions of cotton has frequently been related to the quality of the fiber, especially as regards tensile strength. It has been shown, for example, that chemical treatments which modify the properties of the fibers in such a way as to impair their tensile strength also bring about a lowering of the viscosity in cuprammonium hydroxide

*Research Associates at the National Bureau of Standards, representing the Textile Foundation.

solutions[1, 2]. On the basis of the molecular chain theory for the structure of cellulose this decrease in tensile strength and the accompanying increase in cuprammonium hydroxide fluidity may be regarded as having resulted from a shortening in chain-length of the cellulose molecule. Recently, Farr and her collaborators[3, 4, 5] have brought forth a new explanation based principally on microscopic observations. According to these investigators, the cellulose component of cotton fibers does not dissolve in cuprammonium solution but maintains a visible state of aggregation in the form of diminutive cellulose particles, each of which is surrounded by cementing material which swells in cuprammonium solutions, thereby giving rise to a gel-like structure upon which many of the characteristic properties of the dispersion depend. On the basis of this interpretation it follows that the reduction of viscosity through processing or deterioration of the fibers may be regarded as the result of a direct effect upon the viscosity-producing power of the cementing material.

Since measurements of the viscosity of cuprammonium hydroxide solutions of cotton are widely used to evaluate some of the characteristics of the fiber, and since the above concepts lead to widely different interpretations of the behavior of cotton in this solution, it appeared advisable to undertake a new investigation of the microscopically observable changes which cotton fibers undergo in this reagent.

II. MATERIALS

The cotton used in these experiments was *Gossypium hirsutum* L, Missdel-7 variety, grown at the Delta Experiment Station at Stoneville, Mississippi, and furnished by the Bureau of Agricultural Economics, United States Department of Agriculture. It had been ginned and twice carded, but had received no chemical treatments. In the present paper these fibers are designated as *raw fibers*. The natural waxes were removed from the raw fibers by extraction with alcohol and ether for 24 hours each. These fibers are termed *dewaxed fibers*. A portion of the dewaxed material was further purified by extraction for 6 hours with a boiling 1 per cent solution of sodium hydroxide, according to the method recommended for the preparation of standard cellulose. The procedure was essentially the same as that described by Corey and Gray[6], except that the apparatus of Worner and Mease[7] was employed. Fibers treated in this way were shown by the method of Whistler, Martin and Harris[8] to be free of pectic substance. These fibers from which both wax and pectic substance were removed, are designated *depectinized fibers*.

Two solutions of cuprammonium hydroxide were used in the experiments. One, made according to the recommendations of Mease[9], is essentially the same as that set forth by the Fabrics Research Committee (London)[10] except that the higher concentration of ammonia originally recommended by Clibbens and Geake[11] was used. This solution contained 240 ± 5 g. of ammonia (NH_3), 15 ± 0.1 g. of copper, and 1.0 g. of sucrose per liter. The concentration of nitrite was less than 0.5 per cent. In the present paper this solution will hereafter be referred to as *cuprammonium hydroxide solution A*. A second cuprammonuim hydroxide solution, prepared according to the specification of the Division of Cellulose Chemistry of the American Chemical Society[12] was also used. It contained 30 ± 2 g. of copper, 165 ± 2 g. of ammonia and 10 g. of sucrose per liter. In the present paper this solution will hereafter be referred to as *cuprammonium hydroxide solution B*. When not in actual use, all solutions of cuprammonium hydroxide were kept under nitrogen in tightly stoppered bottles in the refrigerator. Unless stated otherwise, both solutions A and B were used in each of the experiments in this investigation.

III. EXPERIMENTAL PROCEDURE AND RESULTS

Raw, dewaxed, and depectinized fibers were placed, without any mounting medium, on microscope slides, and then examined microscopically as cuprammonium hydroxide solution was drawn under the coverglass. In other cases the fibers were mounted in distilled water, or in ammonium hydroxide, and the cuprammonium solution was added as above. The water and ammonium hydroxide appeared to act merely as diluents, thereby retarding the solvent action of the cuprammonium hydroxide solution. The behavior of the fibers in the cuprammonium reagent was observed microscopically using ordinary and dark-field illumination, and crossed nicols.

Raw cotton fibers, upon addition of the reagent, immediately began to swell and twist, often forming balloon-like structures (Figure 1, A and B). After a few minutes the inner portion of the fiber appeared to be completely dissolved, leaving transparent shells which originally formed the outer surface of the fibers (Figure 1, C). Likewise, a small amount of material from the lumen did not dissolve (Figure 1, B). This behavior was especially striking when observed with crossed nicols. The fibers, originally bright against a black background, became dimmer as the cellulose was dissolved by the reagent. After a few minutes the field was black which indicated that all birefringent material had disappeared. When the same preparation was then examined in ordinary light, it was found to contain many of the shells described above.

Dewaxed cotton fibers presented essentially the same picture as raw fibers. Dissolution of the inner part of the fiber took place in a few minutes, leaving residues (Figure 2) similar to those of the raw fibers (Figure 1, C), but less dense and bulky, and often more torn and fragmented than the latter. With crossed nicols the results were the same as with raw cotton; that is, the strongly birefringent cellulose was dissolved rapidly, leaving an insoluble, isotropic material.

Depectinized cotton fibers dissolved in cuprammonium hydroxide solution, leaving only a very small amount of isotropic residue which consisted of small irregular bits of material (Figure 3). This residue exhibited no definite cytological structure such as that shown by the

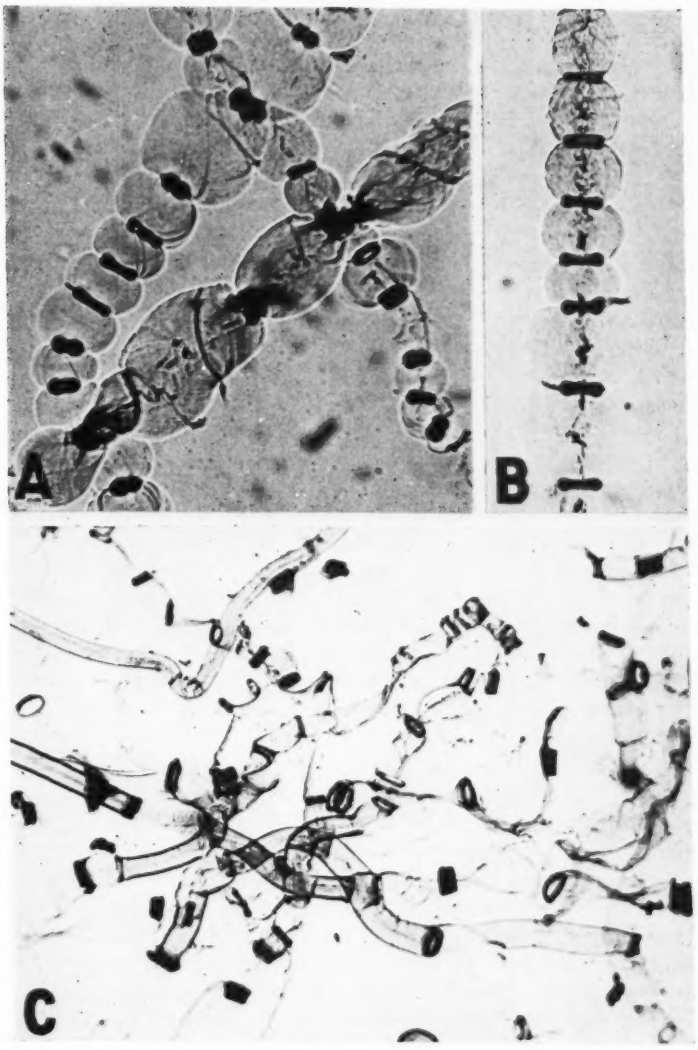
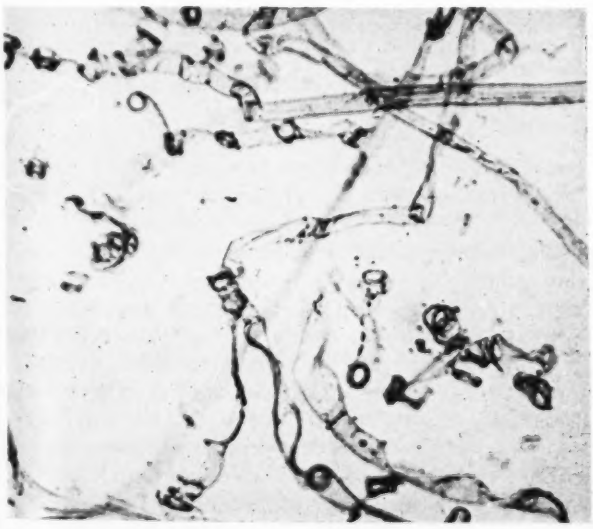
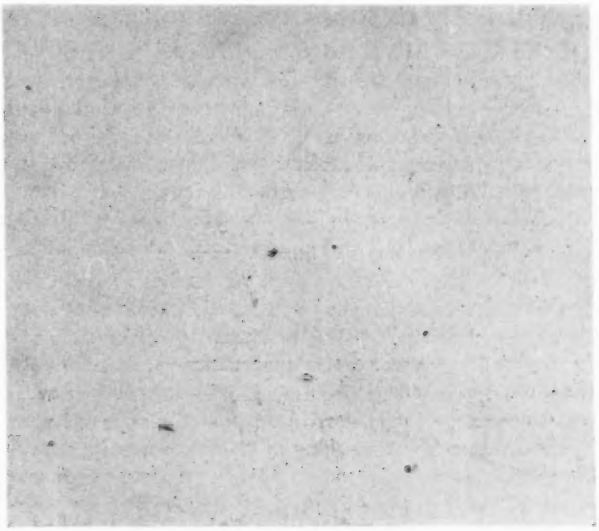


Figure 1 (Right)—Raw cotton fibers in cuprammonium hydroxide solution.
A. Fibers swollen to form "balloons."
B. Typical string of "balloons," showing deeply-stained material in the lumen and at the constrictions between the swellings.
C. Residue which remains after the cellulose of the fibers has dissolved in the reagent. Fibers stained with ruthenium red. Magnification 120 X

Figure 2 (Below, Left)—Dewaxed cotton fibers in cuprammonium hydroxide solution. Residue which remains after the cellulose of the fibers has dissolved in the reagent. Fibers stained with ruthenium red. Magnification 120 X

Figure 3 (Below, Right)—Depectinized cotton fibers in cuprammonium hydroxide solution. Residue which remains after the cellulose of the fibers has dissolved in the reagent. Magnification 120 X





shells of raw and of dewaxed fibers. Compare with Figures 1, C and 2.

The isotropic shells of raw and of dewaxed fibers, and the isotropic material from the lumen did not stain blue when given the iodine-sulfuric acid test for cellulose, but stained deeply with ruthenium red[13]. Although ruthenium red is not a specific stain for pectic substance, native pectic compounds invariably take a red color in the presence of this dye[14].

In the above experiments the effect of cuprammonium reagent on the different samples of cotton could be observed for only relatively short periods. In order to make observations over longer periods, samples of raw, dewaxed, and depectinized cotton fibers were placed in separate vials of cuprammonium hydroxide solution. At intervals thereafter, samples from each of the vials were removed and viewed microscopically, using ordinary and dark-field illumination, and crossed nicols. During the first day the preparations were examined every few hours; during the remainder of the first week, examinations were made once each day, and thereafter once each week for a period of six weeks. After the initial dissolution of cellulose, which appeared to be complete in a few minutes, there was no further change in microscopic appearance of the undissolved material. The solutions of raw and of dewaxed cotton showed nothing but isotropic residues, while the samples containing depectinized cotton were practically free of residue.

These experiments indicate that pectic substance is a constituent of the insoluble shells which remain when cotton fibers are treated with cuprammonium hydroxide solutions. This conclusion is substantiated by direct chemical evidence presented elsewhere[2]. It also appeared from these experiments that the cellulose portion of the fibers dissolved in cuprammonium reagent, leaving no microscopically resolvable particles of the type described by Farr[3], who has stated, however, that the particles are not visible as ordinarily observed since they are obscured to the point of invisibility by a matrix of colloidal cementing material which surrounds them. It is stated further that by maintaining a steady flow of cuprammonium hydroxide over the fibers the matrix may be removed, thereby rendering the particles visible. It might have been expected, however, that the particles would have been visible in the depectinized fibers since these fibers left an almost negligible amount of visible colloidal material after treatment with cuprammonium reagent.

Since this was not the case, it appeared advisable to repeat Farr's experiments exactly as they are described. Raw cotton fibers were mounted in distilled water. The water was then gradually replaced with cuprammonium hydroxide solution by the following procedure: Small drops of the reagent were applied to one edge of the coverglass. The solution was then drawn under the coverglass by means of very narrow strips of filter paper placed at the opposite side. Swelling of the fibers ensued, resulting in the formation of some "balloons" (Figure 1, A and B) which eventually disappeared leaving residues like

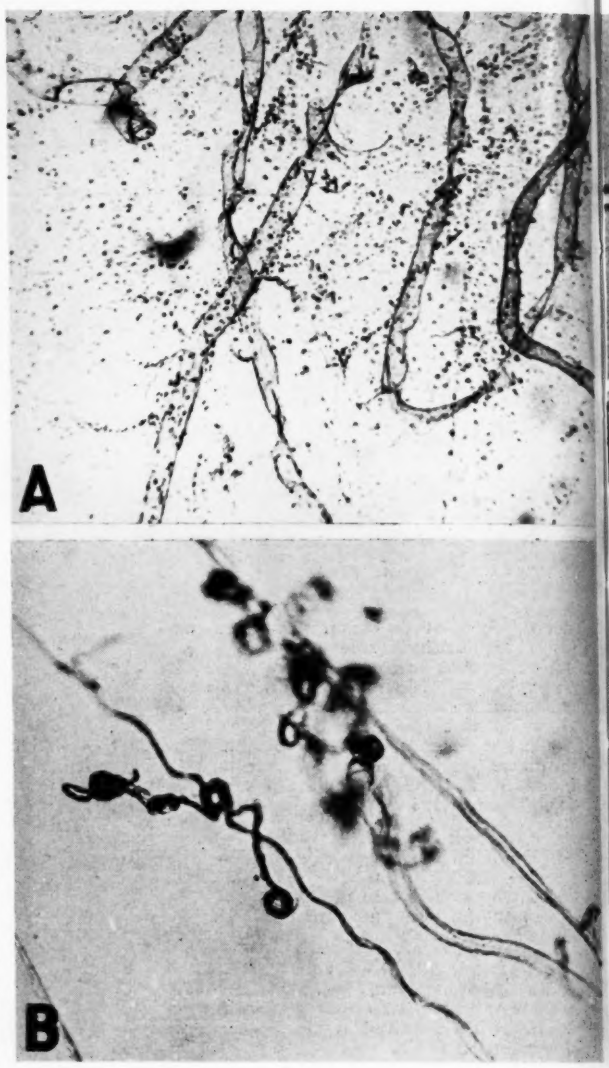


Figure 4—Fibers treated with a steady flow of cuprammonium hydroxide solution.
A. Residue of raw fibers after a continuous flow of the reagent was maintained over them. Particles, formed from the cuprammonium solution, are seen among the residue.
B. Raw cotton fibers after treatment with cuprammonium solution for one hour, under conditions which prevented exposure of the reagent.
Magnification 120 X

those described earlier. However, as the flow of fresh reagent was continued, small particles appeared (Figure 4, A).

The same technique was then applied to depectinized cotton fibers. As previously described, the cellulose appeared to dissolve upon addition of the cuprammonium hydroxide, leaving only a very small amount of insoluble residue. Nevertheless, as with raw fibers, particles became evident when a steady flow of the reagent was maintained.

It was noted, however, that identical particles appeared, even in the absence of fibers, when the steady flow of cuprammonuim hydroxide was continued. The results

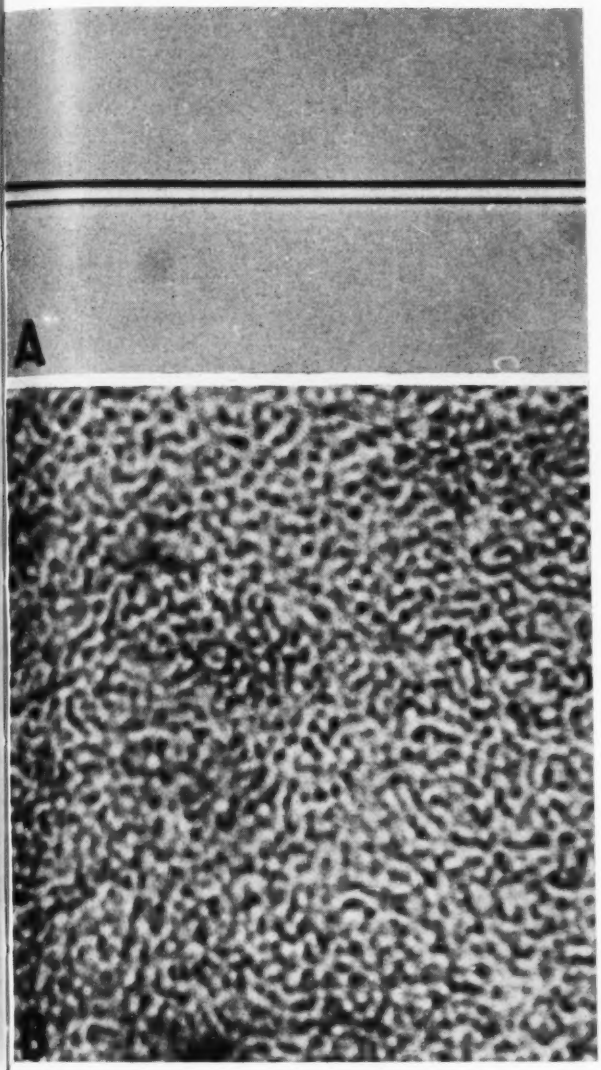


Figure 5—Cuprammonium hydroxide solution: no cellulose present.
A. Fine strand of glass rod in focus in the solution. The latter has not been exposed to allow formation of particles. Magnification 500 X
B. Particles formed upon exposure of the reagent to air. Magnification 1500 X

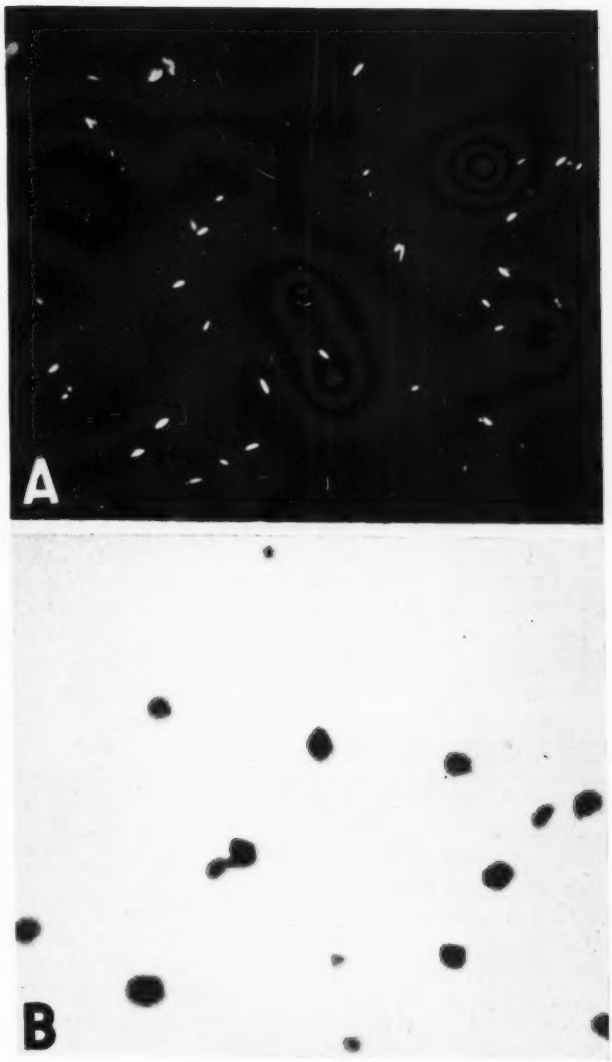


Figure 6—Particles formed in cuprammonium hydroxide solution, in the absence of cellulose.
A. Particles as viewed between crossed nicols.
B. Particles which appear after the addition of iodine and sulfuric acid.
Magnification 1500 X

were the same whether cuprammonium solution A or B was used. In both cases the particles appeared to be ellipsoidal and ranged from 1-2 μ in size. They were birefringent and showed extinction when rotated between crossed nicols. When given the iodine-sulfuric acid test for cellulose, dark particles, somewhat larger than the original ones, were observed. The photomicrographs (Figures 5B, 6A, 6B) of these particles greatly resemble those shown by Farr[3]. All of these photomicrographs are of cuprammonium hydroxide solutions *which contained no cotton fibers*. In all cases the original solutions were clear (Figure 5A), and particles appeared only after the solution was exposed to the air, as in drawing it under the coverglass.

Further examination of the cuprammonium solutions showed that a precipitate was always formed when this reagent was exposed. Farr[3] has stated that a steady flow of the solution had to be maintained in order to prevent crystallization of the copper salt. In the present investigation it was found that regardless of the care exercised in this experiment, particles were always obtained when the cuprammonium solution was allowed to flow under the coverglass according to the described technique.

The experiments in which a continuous flow of cuprammonium reagent was maintained over the fibers were then repeated under conditions which prevented exposure of the reagent to air. In order to do this a sealed mount, constructed as follows, was employed: A ring consisting

of a mixture of paraffin and vaseline was made in the center of a microscope slide, and a few cotton fibers were then placed in the inclosure. A hypodermic syringe was arranged in such a way that the tip of the needle extended just inside the ring, on the opposite side of which a small opening was made. A coverglass was then placed over the fibers and the ring, and further sealed to the slide by applying a viscous solution of methyl methacrylate in acetone, to the edges of the coverglass. The microscope slide and syringe were fastened to a sheet of stiff cardboard so that the whole apparatus could be placed on the stage of the microscope.

The syringe was filled with cuprammonium hydroxide solution. By applying pressure to the plunger of the syringe, the solution was passed slowly over the fibers, which were mounted either dry or in distilled water. The liquid was allowed to flow through the cell and out the small opening in the ring. During this process the behavior of the fibers was continuously observed with the microscope.

Using this specially constructed cell which prevented exposure of the reagent, fibers were treated with cuprammonium hydroxide solutions A and B. It was found that a steady flow of solution A could be passed over the fibers for more than an hour without the appearance of particles (Figure 4B). As the reagent started to flow through the cell the frequently observed reaction took place. The fibers began to swell, thereby giving rise to typical balloon-like structures which eventually appeared to be optically clear. Moreover, as the flow of cuprammonium hydroxide was continued, no particles appeared even after prolonged passage of the reagent.

When solution B was used some particles always appeared even in control mounts which contained no fibers. In all cases in which the solutions were exposed to the air, particles formed more readily with solution B than with A, probably because of the higher copper and lower ammonia concentrations of the former. Because of this fact, solution A was found to be much more desirable for microscopic studies of the behavior of cotton fibers in cuprammonium reagent.

The results of this investigation indicate that microscopically resolvable particles of cellulose do not persist when cotton fibers are dispersed in cuprammonium hydroxide solutions. Particles are obtained, however, even in the absence of fibers, upon exposure of the reagent to air.

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Factors Influencing

Wool Felting

JOSEPH W. CREELY and GEORGE Le COMPTE

A SIMPLE example of shrinking or felting might well be given to afford a clear picture of the phenomena with which these papers will deal. If a piece of yarn is placed in a container with water and vigorously agitated, the yarn shortens. If the agitation is continued for some time and the yarn is then examined, it will be found that in addition to the shrinking, sections of the yarn have begun to grow together with other sections. To produce the original, single, undoubled, unlooped length of yarn, it is necessary to break fibers binding one section of yarn to another. If the adhering sections of yarn are not separated and the agitation is continued long enough, it is possible to produce a mass of wool fiber from which it is impossible to separate a single length of yarn without cutting it out. It is quite evident that to produce this result, fibers from one section of yarn must have entered into intimate contact with fibers from other sections,—there must have been an actual movement and travel of fibers over and around other fibers.

On this characteristic property of wool fibers and properly treated hair fibers are based the felting and fulling processes in use today. A rough, open, unpleasant appearing fabric can be converted by fulling into a smooth fluffy fabric of pleasing appearance. Small tight bunches of woolen noils can be converted into a smooth compact felt. Many fabrics owe their pleasing appearance to the felting or fulling processes.

It is the authors' opinion that the basic phenomena underlying shrinking, felting, fulling, milling, etc., are in all cases the same, and that differences between the

processes are quantitative and not qualitative. We intend hereafter to speak of the general phenomenon as felting, and we feel that the following conclusions are in qualitative agreement with the results that would be obtained in shrinking, fulling, felting or milling experiments. We exclude, of course, shrinking due to stretching a yarn or fabric while wet, and drying it in the stretched condition, so that on subsequent wetting it will spontaneously contract to its normal length.

Many attempts have been made to explain felting phenomena. A good review of these theories has been written by Boxser[3]. However, up to the present, it does not appear that any single property of woolen fibers has been proven to be the prime cause of felting. The presence of scales on the wool fiber might be regarded as an important property necessary for felting. This is clearly shown by the claims of Markert's recent patent[13]. He treats wool with a 1 per cent solution of cellulose acetate in acetone, squeezes, dries and repeats the procedure. A smooth coating of cellulose acetate is formed over the scales on the surface of each fiber. As a result the felting qualities of the wool are greatly reduced. On the other hand wool may be chlorinated so as to retain its scales and still be shrinkproof[9, 10]. Wool shoddies which have had repeated scouring, carbonizing and felting may still exhibit rather good scale structure but no longer have felting qualities. Again, wool that felts very slowly in water can be made to felt quite rapidly in the presence of acids and suitable felting aids.

Apparently then, it appears that the influence of secondary factors may be more important than the inherent felting characteristics of the wool in determining its rate of felting. It is the intention of the writers not to seek for any prime cause of wool felting but to study some of the factors that contribute to the feltability of wool and to try to clear up some of the contradictions found in the literature. Before doing experimental work of our own a search was made in the literature for information pertaining to felting. With the help of this information a list of the factors influencing felting was prepared. It must be pointed out that none of these factors appears to be independent and uninfluenced by other factors.

FACTORS INFLUENCING FELTING OF WOOL

A. ORIGIN OF THE WOOL

Felting characteristics vary with the origin of the wool, e.g., if certain Cape wools are used, the initial shrinking and felting action will be very rapid resembling the properties of a carroted fur fiber. As soon as this initial shrinkage has taken place further action produces but slight additional felting. If, on the other hand, a Texas wool is used, it will be found that the initial shrinkage and felting is much slower but after continued fulling for a long time it will be found that the fibers continue to felt until an almost rock hard felt is obtained. According to Hayes[11] the order of felting ability of wools is as follows:

Fine Texas Wools
Fine Cape Wools
Fine Australian Wools
Fine California Wools
Fine Territory Wools

In addition to the peculiarities exhibited by various types are the added characteristics of climatic conditions, feeding, time of clip, etc., which also influence felting qualities.

Nitschke[15] has shown that the addition of viscose, cuprammonium, acetate rayon and lanital staple fibers to wool before felting in general gives felts having lower tear factors.

B. PHYSICAL PROPERTIES OF THE FIBER

1. *Fineness*—It has been stated that a fine wool in general felts better than a coarse one[21].

2. *Fiber Length*—There is some disagreement on this point in the literature. Thus, Speakman, Stott and Chang[21] state that Wensleydale wool of inferior scaliness and longer staple is superior in felting qualities to the finer and more scaly Southdown wool. On the other hand Boxser[3] states that fine wool of short staple usually felts best.

3. *Scaliness*—It has been shown that the more scaly wools in general felt better than less scaly wools[20,21].

4. *Crimp*—A crimpy wool felts better than a non-crimpy wool, other things being equal[20].

5. *Rigidity of fiber*—It appears that flexible wool fibers felt better than the more rigid ones.

6. *Elasticity*—Ability of wool fibers to stretch and recover from extension is probably an important factor necessary to felting. Speakman, Stott and Chang[21] have shown that there is a loss in the power to recover from extension beyond 45° C., corresponding to a maximum felting action as found by them at around 45° C.

C. PREVIOUS CHEMICAL TREATMENT OF THE FIBER

1. *Oxidation*—Many chemical treatments that must be regarded as oxidizing are applied to wool, hair and fur fibers. It is surprising to discover that very similar treatments are at times intended to decrease the felting properties and in other cases to act as carrotting treatments and increase the felting properties.

(a) Hydrogen peroxide—Treatment with this material in acid solution seems in all cases to result in enhanced feltability of the fiber. Thus Brown[5] has described a process used in Germany for increasing the feltability of wool in which the wool is treated for some 6 to 8 hours with a 0.15 per cent solution of hydrogen peroxide at a pH of 1 to 2 in the presence of a metal catalyst. Brauckmeyer and Rouette also describe methods for increasing felting power with hydrogen peroxide[4].

(b) The halogens and many of their derivatives are widely used to decrease the feltability of wool. Chlorine, bromine, iodine, hypochlorous acid, sodium hypochlorite[22] and alkyl hypochlorites[17] may be used in the shrinkproofing of wool. Sulfuryl chloride, SO_2Cl_2, has also received considerable publicity as a shrinkproofing agent[10]. It is sometimes classified as an oxidizing agent of the same type as the halogens[19] although such properties seem somewhat at variance with the chemical constitution of sulfuryl

chloride. Very surprisingly, a large number of patented carrotting compositions seem quite similar to the materials used above for shrinkproofing. Thus, the following are used as carrotting compositions:

a. 100 parts water
3 parts mercuric sulfate
9 parts hydrochloric acid
4 parts sodium peroxide[8]

b. 100 parts water
5 parts sulfuric acid
1 part hydrochloric acid
3 parts potassium chlorate
4 parts hydrogen peroxide
15 parts sodium sulfate[7].

It seems that such baths would necessarily contain some free chlorine. Böhm[2] claims that he can render cross-bred wools fit for felting by soaking in an aqueous solution containing 0.05-0.2 per cent hydrochloric acid and 0.25-1 per cent hydrogen peroxide for 30 minutes. After removing the excess of liquid the hair or wool is thoroughly dried at 70-100° C. This bath would certainly contain some free chlorine.

The obvious contradiction in results obtained with similar shrinkproofing and carrotting preparations should be fully investigated to determine the conditions under which the two actions can be made to take place.

(c) Other oxidizing solutions are intended to increase the feltability of animal hair. Thus, a solution of an oxidizing agent containing a small amount of an oxygen carrier and a large amount of a metallic compound which can be adsorbed by the fiber is used to increase feltability. As a definite example of this general formula, Polgar[16] gives:

180 mls. nitric acid
180 gms. dry aluminum sulfate
25 gms. potassium chlorate
2 gms. of a salt of vanadium
Water to make a liter of solution.

2. *Alkaline Treatment*—Alkaline materials such as borax, soda ash, and sodium hydroxide are reported to decrease the feltability of wool[22]. Also it is reported that pulled wools are inferior to fleece wools in felting[11]. Alkalies are also used in carrotting solutions to increase the feltability of wool and hair. Preliminary work by the authors indicated that treatment with borax, soda ash or sodium hydroxide increases the rate of felting of woolen yarns. This conflict in the reports of the results obtained with alkaline treatments indicates the need for investigation.

3. *Treatment with Acids*—Acid materials are reported to decrease the feltability of wool. Thus Hayes[11] reports that the treatment of wool under normal carbonizing conditions decreases its shrinkage. On the other hand the treatment of wool with 10 per cent sulfuric acid and 2 per cent glycollic acid has been patented as a carrotting treatment[14]. Apparently here, too, is a field requiring further study.

4. *Treatment with Reducing Agents*—

(a) Pulled wool is said to have poorer felting qualities than fleece wools. Alkaline sulfide solutions are usually employed in the pulling operation.

(b) Jones has patented the use of alkali sulfites for decreasing the felting capacity of wool[12]. He recommends a pH of 8 and temperature of 70-100° C.

(c) Von Bergen and Mauersberger[23] claim that treatment with 4 per cent formaldehyde reduces the felting capacity of wool.

D. PHYSICAL PROPERTIES OF THE FELTING SOLUTION

1. *Lubricating Properties*—The lubricating properties of the felting solution are probably an aid to felting. Soap solutions which possess some lubricating qualities felt more rapidly than sodium carbonate solutions of the same or even higher pH[21]. Wool may be fulled in the presence of a dispersion of a neutral lubricant in water[1,6]. Wool also exhibits an increased tendency to felt in the presence of certain wetting agents in an acid solution. This is probably due to lubrication of the wool.

2. *Viscosity*—Fulling solutions are sometimes made up with high titer soaps which give highly viscous solutions. These solutions are preferred by the trade to the less viscous low titer soap solutions.

3. *Presence of Moisture*—It appears that wool will not felt in non-aqueous media. It may be that the moisture is necessary to make the scales project and exert an influence on felting. Wet fibers show a higher difference in the coefficient of friction between the root-tip and tip-root directions than do dry fibers[21]. Berg[1] has described fat fulling of fabrics, in which the fabric is fulled in the presence of a mixture of fat and water. In this process it is essential that the water be present within very definite limits. In ordinary felting practice excess water has been found to decrease rate of felting; if the material is too dry there is a tendency toward chafing and fiber damage.

4. *Temperature*—Temperature has a considerable effect on the rate of felting. Speakman, Stott and Chang[21] found a maximum in the fulling of woolen cloth at a temperature of about 45° C. There is some conflict on the subject of the effect of temperature on felting. Schofield[18], basing his conclusions on the production of felt from woolen stock in a Bywater machine, and on the density, porosity and strength of the felts produced, concluded that felting as measured by that method was increased by temperature up to 98° C. On the other hand, there is some indication in Schofield's own data of a maximum action around 45° C. We include the following data from Schofield[18]:

Temperature of Felting, °C.	*Dry weight of 280 Sq. inches of felt*
18	2.03
37	2.23
46	2.28
55	2.15
65	2.18

(Concluded on page 306)

PROCEEDINGS OF THE
American Association of Textile Chemists and Colorists

JUNE 10, 1940

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• CALENDAR • OF COMING EVENTS

Outing, Rhode Island Section, Metacomet Golf Club, Barrington Parkway, East Providence, R. I., June 14, 1940.

Outing, Philadelphia Section, Seaview Country Club, Absecon, N. J., June 15, 1940.

Outing, Piedmont Section, Ocean Forest Hotel, Myrtle Beach, S. C., June 21 and 22, 1940.

Outing, Mid-West Section, Lake Lawn, Delavan, Wis., June 22, 1940.

Annual Meeting and Convention, auspices New York Section, October 18th and 19th, 1940, Hotel Commodore, New York City.

The Treatment of

Spun Rayons with Textone*

G. P. VINCENT, A. L. DUBEAU and J. W. IVEY**

TEXTONE, as you undoubtedly know, is essentially sodium chlorite, $NaClO_2$. This is a commercially new oxidizing agent and has recently been made available by our company. Information on this product was originally given in a paper at the Boston Meeting of the A.A.T.C.C., on September 15th, 1939. The paper was published, following this meeting, in the DYESTUFF REPORTER for October 2nd, 1939, under the title "A New Oxidizing Agent for the Treatment of Textiles." A general discussion regarding Textone was also given by a member of our staff at the A.A.T.C.C. Meeting in Greenville, S. C., on February 10th, 1940. This talk was also published in the DYESTUFF REPORTER on May 27, 1940. Both of these papers outline the general properties of Textone and mention briefly the general applications which are being made in the textile industry. These consist of addition of the material to kiers for the purpose of obtaining a better "bottom," the elimination of greige acid souring, the elimination of multiple kier boiling, or as an aid in proper desizing. Information was also given in these articles on the treatment of spun rayon with Textone in conjunction with a synthetic detergent for the purpose of scouring and bleaching in one operation. This process is also applicable when bleaching is not necessary as is the case in preparing spun rayons for dark shade dyeing. Minor uses mentioned include the use of Textone in obtaining more even mercerization, stripping colors and preparing soluble starch.

Tonight we would like to limit our discussion almost exclusively to spun rayon treatment. Satisfactory preparation of spun rayon is, of course, being accomplished today in all mills throughout the textile industry by conventional processes ranging from kier treatment to treatment in jigs. It has been our experience, however, that most mills are not completely satisfied with the simplicity, cost and excellence of results obtained by these well known methods.

The use of Textone offers a finishing plant the opportunity of reducing processing time and obtaining a superior result at no additional cost and very often at a reduced cost. Treatment of spun rayon with Textone falls broadly into two different classes; namely, those fabrics which are to be bleached and left in this condition without dyeing and those which will be dyed. Strictly speaking, dyed fabrics can be further subdivided into those being dyed dark shades which, as a usual thing, will require no bleaching, and those being dyed light or pastel shades where a small amount of bleaching is desirable.

FULL BLEACH—MODERATE WHITE

Spun rayon will usually be sized with starch or gelatin. If the goods are sized with starch, no special desize treatment need be given unless a particularly soft hand is desired. If this is the case, treatment with any good desizing agent is satisfactory. If the size is gelatin, the goods should be given a hot water wash as the first step. This solubilizes and removes the gelatin. If this is not done the gelatin may precipitate as a gum in the acid Textone bath.

After the desize treatment, if any is necessary, the spun rayon is treated in a bath containing acid, Textone and synthetic detergent. In this manner a full bleach and complete scouring are obtained in one operation. A Typical example is as follows:

Equipment: Dyebeck—Capacity 1,700 gallons
Cloth: Spun Rayon—3,000 yards
Water—1,700 gallons.
Textone—3 to 4 pounds
Detergent—2 pounds.
Acetic Acid, 28 per cent—6 to 10 quarts.

The order of addition of the above compound to the solution is unimportant. After the solution has been prepared and the goods are in the solution, the temperature is increased to as close to the boiling point as possible. The goods are run through this solution until the whiteness desired is obtained. This will usually be accomplished in an hour. At this point, the liquor is dropped and the spun rayon is given a hot water wash. This can be followed with a cold water wash, but is not necessary. No antichlor is needed. The operation is complete with the last wash. Some types of spun rayon may require more than the hour mentioned above.

FULL BLEACH—EXCEPTIONAL WHITE

A white superior to any probably being attained at present results from a two step process, employing Chemic in the first step followed by the Textone formula given above. If necessary the goods are desized and washed. They are then treated with Chemic by one of the two following methods:

Chemic Method I—Padding

Pad on a 3 degree Twaddle Chemic solution and allow the goods to lay for 2 hours. Then give them one cold water wash to remove part of the

*Presented at meeting, Southeastern Section, May 11, 1940.
**Mathieson Alkali Works, Inc.

Chemic. The goods are then ready to be treated in the Textone formula previously outlined.

Chemic Method II—Continuous

Treat in a dyebeck or Rodney Hunt kettle, using one degree Twaddle Chemic solution. The retention time in this equipment should be twenty to thirty minutes. A single cold water wash is then employed to remove a portion of the Chemic and the goods are then treated in the Textone formula previously outlined.

One need not fear tendering from residual Chemic as the goods enter the hot, acid, Textone solution. This is due to a specific reaction between Chemic and Textone which prevents any degrading action on the cellulose.

It is necessary to emphasize a precaution regarding metals which may be immersed in the bath because of their effect on color. Stainless steel, nickel and wood have been found satisfactory. The presence of even a small amount of copper, bronze or brass, is detrimental. Sufficient corrosion occurs so that the cloth obtains a slight greenish or yellow tint. A Rodney Hunt kettle, dyebeck, or similar equipment, is satisfactory to use in this process as long as attention is given to the metals of construction. We wish to emphasize that even though the main portion of the machine may be made from wood, stainless steel, or nickel, for satisfactory results no roll or any other small item in the equipment should be made of copper, brass or bronze if it touches the solution or cloth. If however one wishes to try this process with small amounts of copper, brass or bronze present the addition of 0.25 lbs. of tetra sodium pyrophosphate per 100 gal. of solution will reduce corrosion. This would be 4.25 lbs. in the 1,700 gal. batch previously mentioned. If the phosphate is used some additional acid may be required to overcome the buffering effect of it.

ROPE PREPARATION OF SPUN RAYONS TO BE DYED

As stated previously, spun rayons to be dyed can be classified into those receiving dark shades and those receiving light or pastel shades. In conventional methods of preparation these two types of fabrics may or may not be treated differently. In most mills the fabrics dyed in dark shades will be given no bleaching treatment. In some mills, the spun rayons to be dyed light shades will also not be bleached in any way, but in other mills a light bleach may be applied. In treating fabrics to be dyed with Textone, we treat all of them essentially the same for reasons which we hope will be made clear to you later in this discussion and, consequently, we will make no distinction between the two types.

It is, of course, necessary to scour these fabrics in order that the dye be taken up evenly by the cloth. Excellent results have been obtained by using the combined Textone-synthetic detergent bath in an acid solution. In batch treatment a typical example, modeled after the one given above for full bleaching, is as follows:

Equipment: Dyebeck—1,700 gallons
Cloth: Spun Rayon—3,000 yards
Water—1,700 gallons
Textone—1 to 1½ pounds.
Detergent—2 pounds.
Acetic Acid, 28 per cent—6 quarts

Previous to treatment in the above bath, the goods should be desized if desired, according to the outline we have mentioned for spun rayons being given a full bleach. Treatment in the above solution at a boiling temperature for approximately one-half hour is sufficient. The liquid is then dropped and the goods given a hot water wash followed by a cold wash, if desired. They are then ready for dyeing.

A continuous process can be made of this operation by using dyebecks or Rodney Hunt kettles in tandem. Approximately, three pieces of equipment would be necessary. Hot water is placed in the first kettle. The second kettle is filled with water and as an example, we cite 600 gallons. To this is added 2 pounds of Textone, 2 pounds of detergent and about 3 quarts of 28 per cent acetic acid. The third kettle contains hot water. The second kettle containing the chemicals is maintained at a boiling temperature. The goods are run continuously through those baths and unloaded through a squeeze roll and dyed or dried. In this process the water should be changed frequently in kettle No. 1 to eliminate the possibility of seriously contaminating kettle No. 2. The concentration of the Textone in the second kettle should be maintained by adding approximately one pound of Textone for every 1,000 yards of goods treated. Likewise, a synthetic detergent concentration is maintained by adding approximately ½ pound of detergent for every five or six thousand yards of goods going through the solution. Occasional additions of acetic acid may be necessary to maintain the bath on the acid side. After fifteen or twenty thousand yards of goods have been treated the solution in kettle No. 2 containing the chemicals should be dropped and a fresh solution made up.

In this process the goods can be treated at a rate of about 3,000 yards an hour. The actual consumption of chemicals is low and the cost per yard is very satisfactory. The dyeing results obtained are excellent with a minimum consumption of dye. The hand of the goods is preferable to that ordinarily obtained, due to the fact that the operation is short and the fabric is kept on the acid side. The short treatment time resulting in less handling of the goods is an attractive feature, from the standpoint of preventing physical distortion of the weave. All spun rayons which are to be dyed can be treated by the batch or continuous processes outlined above, with results which, in our experience, cannot be excelled. This applies to both light and dark shades. If a mill is attempt-

ing to obtain the best possible effect and wishes to be assured that the quality of its work is unsurpassed, the use of these processes will accomplish this.

OPEN WIDTH PREPARATION OF SPUN RAYONS TO BE DYED

If a mill, from desire or necessity, wishes to reduce its cost below that obtaining in the processes mentioned, the process which we are about to describe offers this possibility. The results obtained will be good; in fact, during the majority of the time the results may be very nearly equal to the previous processes outlined. A different type of equipment is necessary, such as a continuous open soaper and a very large quantity of goods will have to be processed in order that the minimum cost be obtained. Below is a typical example:

If the goods contain no acetate rayon, a desizing operation, if necessary, can be carried out by padding on a 1½ to 2 per cent caustic solution at a temperature of 100° to 120° F. The goods are batched in rolls and let stand for 4½ to 5 hours. It is not necessary to desize in this manner, ordinary desizing operations being satisfactory, except the use of the caustic may slightly reduce the cost. In the case of acetate rayon goods, it is of course necessary to use desizing agents and not caustic. After the desizing operation the goods are continually run through an open soaper or similar equipment in full width. Such an apparatus containing seven compartments with 600 gallons of solution in each compartment, is as follows:

1st compartment—boiling water or water containing acid to neutralize caustic.
2nd compartment—hot water—3 quarts, 28 per cent acetic acid.
3rd compartment } Each: 2½ lbs. Textone
4th compartment } 2½ qts. Acetic Acid Temp. at least 200° F.
5th compartment } 3½ lbs. detergent
1 lb. Tetra sodium pyrophosphate
6th compartment—hot water, 2 lbs. synthetic detergent.
7th compartment—hot water.

The goods are then mangled in cold water and are ready for dyeing. If the open soaper contains more than seven compartments, additional washing can be resorted to in the last compartment. It may be necessary at the start to use one compartment for caustic and hydrosulfite to remove fugitive colors, especially reds and blues.

The materials of construction of the compartments must follow the principles mentioned before.

The speed of this process is great. The cloth is in any individual solution for only a matter of seconds. If large yardages are to be run, the consumption of chemicals is extremely low. Because of this and the low labor cost, the process is inexpensive. The total cost should not be greater and will, in all probability be less, than any process being used for treatment of similar goods. The result, in our experience, has always been superior to conventional processes in evenness of dye penetration, brightness of shade, hand, and general customer appeal.

GENERAL DISCUSSION AND SUMMARY

We would like to make some further remarks regarding the use of these processes in the preparation of spun rayons to be dyed. It usually occurs to the man in charge of a finishing plant that if the shade is such that no bleaching is necessary, the use of an oxidizing agent is an added and unnecessary expense. This is a very logical way for him to reason. It is the way that we, ourselves, reasoned in the beginning of our development work and it took a great deal of experimentation before we were thoroughly convinced that this was not the case. It is necessary, previous to dyeing, to scour the goods as a minimum treatment. The addition of Textone to the bath does not increase the number of operations. Consequently, the time factor does not enter into the cost by using Textone. Scouring, previous to bleaching, is done in order to assure a good dyeing result. We ordinarily think of this as the removal of materials which lend themselves easily to emulsification or suspension, known to the colloid chemist as peptization. The synthetic detergents alone, or any scouring agent you are now using, will undoubtedly accomplish this. It will remove from the vicinity of the fiber, extraneous dirt and non-cellulosic impurities not intimately connected with the physical structure of the fiber. Such materials, however, are not the only ones which have an effect on dye penetration and evenness of dye adsorption.

Textone, fortunately, is not decomposed rapidly by acid or temperature. It, likewise, does not react with synthetic detergents to injure the detergent's cleansing properties. This non-reactivity is mutual and the detergent does not injure the oxidizing properties of the Textone or its stability. This is a happy state of affairs and would be difficult to attain with almost any other oxidizing agent. This means that the bath is exceedingly stable. The small amount of Textone used results in slightly bleaching the goods which removes from the fiber non-cellulosic material which is beneficial to good dyeing. This is not concerned with the whitening of the fiber, but with the removal of impurities.

An analogous case presents itself in the preparation of cellulose from wood for paper manufacture and rayon production. The process resorted to in some respects resembles the scouring of textiles and their subsequent bleaching. The cellulose is violently cooked in various solutions under pressure for a long period of time in order to remove the lignin or non-cellulosic bodies. After this, the pulp is treated with chlorine and hypochlorite to bleach it. This, however, is considered a purification process for the bleaching operation results in the removal of lignin. In many pulps after the cooking operation, the cellulose contains as much as 15 per cent lignin. The bleaching operation not only makes the pulp whiter, but it also removes this lignin material and, if handled in the right way, cellulose can be made from wood which contains 98 to 99 per cent alpha cellulose. The manufacturer of

this product bleaches not only to obtain the color desired, but also to purify the pulp in a way so that it reacts chemically as almost pure cellulose. This is the result you desire to achieve in preparing a fabric for dyeing and the more impurity which can be removed from the cellulose of the fabric, the better will the dye job be. That is the point we are trying to make. If you can purify the cellulosic product with which you are dealing to a higher degree than you are doing now in present processes and if this purification job costs you no more, you will be accomplishing a worthwhile and progressive modification.

The various formulas and procedures mentioned in this paper have been conceived with the above idea in mind. Careful attention has been given to cost and it is believed that you will find, upon investigation and experimental study, that your process of treating spun rayon can be adapted to one of the ones we have outlined which will result in a more satisfactory fabric at a cost no greater, and possibly less, than your present operation. The bleaching action which results, as distinguished from the purification or removal of non-cellulosic materials, is an added advantage in the production of brightness of shade. The processes are designed so that the goods are scoured with the Textone bath at approximately the same cost you are undoubtedly obtaining now. We have attempted to show how the Textone oxidizing action removes impurities and gives better dyeing. The bleaching which accomplishes this is, if one chooses to look at it in such a way, a simultaneous result not imperative to good dyeing, but very beneficial in the production of brighter shades.

Before leaving the subject of the effect of the Textone on the fiber, we also wish to point out that besides removing impurities which may occur naturally in the fiber, Textone also has an effect on materials such as starch. We would not recommend the use of acid Textone in this bath for a desize. In those cases where desizing is necessary, if the starch is not removed completely or if it is not removed from all portions of the fabric to exactly the same extent, some unevenness in dyeing may result. The solubilizing action of the acid Textone solution on the starch is sufficient to remove the remaining traces of starch or even the starch distribution in such a way that no unevenness in dyeing occurs.

Another very definite advantage is concerned with hand. It has been our experience that the hand obtained in this process is not being obtained in conventional processes at this time. We believe that this is due to the processing of the fabric entirely on the acid side. If caustic impregnation is used for desizing the hand may be somewhat harsher than if desizing were accomplished by other means.

It will be apparent from a study of the processes mentioned that the fabrics are given a minimum of handling. It is unnecessary to emphasize the desirability of this feature and its beneficial effect on the structure of the fabric. You are all familiar with the necessity of this, particularly in the handling of spun rayons.

We believe that this discussion indicates a better way of processing spun rayons which are to be fully bleached. We also trust that we have outlined processes covering the needs of specific mills who are handling spun rayons to be dyed in small or large quantities. We hope that using the principles mentioned and adapting them to your own particular plants and needs will be of benefit to you.

MEETING, SOUTHEASTERN SECTION

THE spring meeting of the Southeastern Section was held on Saturday evening, May 11, 1940, at the Hotel Ralston, Columbus, Ga. Perrin N. Collier, Vice-President and Director of Research of Callaway Mills, LaGrange, Ga., who is the chairman of the Section, presided. The meeting began with a dinner, and the program that followed contained, in keeping with the program policy of the Section, one technical paper and an address of a general nature on a subject of interest to the group.

J. W. Ivey, of Mathieson Alkali Works, Inc., Charlotte, N. C., presented a technical discussion of "Textone," the new oxidizing agent developed by his company and first announced at the National Convention of A.A.T.C.C., in Boston in September. In his talk at Columbus, Mr. Ivey concentrated upon the uses and applications of his material in connection with the finishing of fabrics containing spun rayon yarn. There was a brief round table discussion of the subject at the conclusion of his address. B. J. Kane, Purchasing Agent, Fulton Bag & Cotton Mills, Atlanta, Ga., was the other featured speaker, discussing "Cotton Goes to Market," and giving the members of the Section an interesting and informative description of the growing, marketing, grading, classification, etc., of cotton.

The Program Committee of the Section is composed of A. Kempton Haynes of Rohm & Haas Company, Inc., Atlanta, Ga.; C. Russell Gill, of Southern Sizing Company, Atlanta; and Robert W. Philip, Editor of *Cotton*, Atlanta.

A guest at the Columbus meeting was Dr. Walter M. Scott, Chief of the Cotton Chemical Finishing Division of the Southern Regional Research Laboratory being established at New Orleans, and chairman of the Technical Program Committee for the annual convention of A.A.T.C.C., in New York in October. Dr. Scott spoke briefly, outlining broadly the plans of his new work at New Orleans and, as chairman of the National Technical Program Committee, described the proposed session for the New York convention to be devoted to technical papers presented by the various sections of the association.

L. L. Bamberger, Vice-Chairman of the Southeastern Section and Chairman of its Membership Committee, reported briefly on the activities of his committee, pointing out that the Section enjoys the largest membership in its history, having now more than 160 members.

There were 131 members and guests in attendance at the Columbus meeting.

Respectfully submitted,

CURT MUELLER, *Secretary*.

MEETING, MID-WEST SECTION

A MEETING of the Mid-West Section was held at the Schroeder Hotel, Milwaukee, Wisconsin, on Saturday, May 4, 1940.

At 3:00 P. M., Archie Alexander called the meeting to order. Mr. Alexander introduced Paul Logue of the Monsanto Chemical Company. Mr. Logue gave an illustrated talk on "Some Uses of Tetro Sodium Pyro Phosphate in the Textile Industry." The meeting adjourned at 4:00 P. M. Dinner was served at 7:00 P. M. There were 89 members and guests present.

The business meeting was called to order at 9:00 P. M. by Vice-Chairman Archie Alexander. The Secretary's report was read and approved. Mr. James Morrison gave the outing committee's report regarding possible sites for the annual outing. Members decided to hold the outing at Lake Lawn, Delavan, Wisconsin, on June 22, 1940. Chairman Herman Boxser announced that there is a prize for the best paper presented at the National Convention in New York this fall.

Mr. Alexander then introduced Norton Thomas, City Chemist and Bacteriologist. Mr. Thomas presented an illustrated talk on the "Purification of Milwaukee's Water Supply." This proved very interesting and was well received.

A film was then run, showing package dyeing in the Wiscassett Mills, Albermarle, N. C. This was an excellent film and was made by Thomas Smith, the dyer at this mill.

The meeting was adjourned at 10:30 P. M.

Respectfully submitted,

DAVID A. ANDERSON, *Secretary.*

LAPEL BUTTONS

These buttons, bearing the seal of the Association, can be purchased by members in good standing from the Secretary at $1.50 each.

UNEMPLOYMENT REGISTER

Members of this asscciation, who are without employment, should forward to the secretary a statement as to their qualifications and experience. This information will subsequently appear on these pages with an identifying key number. Employers are also requested to file with the American Dyestuff Reporter any vacancies which may occur in their business.—H. C. Chapin, Secretary, Lowell Textile Institute, Lowell, Mass.

NOTICE TO EMPLOYERS

Complete personal histories and employment records of the following applicants are on file at the office of the American Dyestuff Reporter.

Key: A—Laboratory and research; B—Dyeing or printing; C—Finishing; D—Sales; E—Instruction; F—Executive.

NOTE: PERSONS WHO HAVE OBTAINED EMPLOYMENT SHOULD NOTIFY THE SECRETARY OR THE AMERICAN DYESTUFF REPORTER AT ONCE.

A-B-7

Education—Evening school in textile dyeing and chemistry.

Experience—Dyehouse laboratory for 2 years. Foreman dyer and finisher on hosiery for 9 years. Experience on ribbons also vat dyes on rayon and silk skeins. 2 years assistant to superintendent. Desires position either in demonstrating and sales or dyer; New York Viciniy. Age 33; married.

A-B-8

Education—Graduate Chemist.

Experience—Print superintendent, colorist; eleven years' experience in all types of fabrics, specializing on rayon; 8 years' dyestuff laboratory and demonstrating. Excellent references; age 37; married.

A-B-9

Education—Bradford Durfee Textile School, 1918, chemistry and dyeing course.

Experience—Dye application, textile and color chemist; good general factory, laboratory and office experience; accustomed to assuming responsibility; handled all types of help; purchased chemicals, dyes, etc.; executive ability; good personality.

Age 42; American, married; will go anywhere for interesting work; references.

A-B-10

Education—1 year of textile chemistry, day course, Philadelphia Textile School; also business course.

Experience—3 years as apprentice in weaving mill in Germany, office and stock room, general clerical work, practice in all departments of cotton manufacture, dyeing, bleaching and finishing of cotton-rayon mixtures, piece and package processing; several mos. in laboratory of German dyestuff manufacturer, dyestuffs and assistants and their application, vat and naphthol dyeing.

Age 20; will go anywhere in U. S.

A-B-11

Education: Graduate chemist. Correspond and converse German, some French.

Experience: 3 years dyer; 4 years dyestuff testing, trouble-shooting, purchasing; 4 years demonstrating, consulting, and supervising for large dyestuff manufacturer. Thoroughly experienced on rayon, cotton, silk, acetates, printing, vats. Fullest references as to character and ability. Seeks position as technician, consultant, or superintendent with reliable dyestuff sales organization or dyehouse. Age 33, married.

A-B-12

Education—Philadelphia Textile School (Evening), Chemistry and Dyeing, Quantitative and Qualitative Analysis.

Experience—Two years in yarn office, making wool stock blends. Last 6 years in dyehouse, 4 years as kettle hand, 2 years in piece dye work. Worked on yarn, raw stock and piece goods; has been dyer on union piece goods, cotton, rayon and wool, leaving wool white; also worked on all wool fabrics and woolen cross dyes; also considerable laboratory experience doing laboratory dyeings and extraction work. Seeks position as dyer or laboratory technician.

Age 30; married; references.

A-B-13

Education—B.S., Chemical Engineering, Oregon State College, 1926.

Experience—First experience in dry cleaning plant; few months as sampling and control chemist with potash and chemical company; two years in dry cleaning plant, all phases of work; few months on routine and special analyses, plant problems, special studies and research for gas and electric company; 10 years teaching experience on textile chemistry, dyeing and textile testing covering all general and special courses in this field; consultant work with various mills. Seeks position in textile chemistry or dyeing.

Age 37; married; will go anywhere; references.

A-B-14

Education—Graduate of New Bedford Textile School, chemistry and dyeing course.

Experience—Two years in dyeing filament rayon skeins; seven years general laboratory work in large print works; four years overseer of package dyeing of cotton and spun rayon with all types of colors. Plant liquidating; must find other employment

Age 34; married; references.

A-B-C-4

Education—Educated abroad.

Experience—Twenty years experience in the textile field as bleacher, dyer, finisher and chemist. 16 years in charge of dyestuff laboratory of large dyeing plant, 4 years as chief chemist with large bleachery. Has tested dyestuffs, and chemicals used in the textile field and has manufactured soaps and textile specialties. Has handled all textile fibers; bleaching, printing, dyeing and finishing same. Has done pigment and lacquer printing, waterproofing and fireproofing. Reason for leaving last position was due to business conditions.

Age 41; married.

A-B-C-8

Education—B.T.C., 1933, Lowell Textile Institute.

Experience—5 months as rayon slasher tender; 8 months color matching, dye testing and analytical work; 5 years with large print works in rayon finishing department, laboratory, color matching, dye testing, assistant rayon dyer. Seeks position as rayon dyer, assistant dyer, or any job in a rayon dyeing and finishing plant. Age 29; single.

A-B-C-9

Education—Lowell Textile Institute; chemistry, dyeing and textile finishing.

Experience—Twelve years head dyer in hosiery mills. Dyeing all types of fibers and in combination. Knowledge of all dye applications. Matching and formula making. Charge of every department in a hosiery mill except fixing machinery. Has done jigg work on cottons and rayons. Desires position in hosiery, or in any field. Would like to work as an assistant to head dyer in a woolen or rayon mill. Age 42; married. Will go anywhere in the United States.

A-B-C-D-1

Education—Lowell Textile Institute, Bachelor of Textile Chemistry; Lowell Textile Institute (Evenings), 2 years D. C. Electricity; 2 years A. C. Electricity.

Experience—Six years as chemist and assistant dyer with a large organization. Specialized in dyeing, finishing, bleaching and drying of woolens, worsteds, cottons, rayons and unions in the piece. Some sales experience. Desires position as chemist, dyer, finisher, bleacher or sales. Age 30; American citizen; Married.

A-B-C-E-1

Education—3 years Chemical Engineer major in college; honor graduate Philadelphia Textile School, chemistry and dyeing course.

Experience—Ten years experience as chemist, colorist, technical director in bleaching, dyeing and finishing of cottons and rayons. Also one year's similar experience in South America. Instructor of textiles and textile chemistry in state vocational school, 2 years. Married; age 32; references; will go anywhere.

A-B-C-F-1

Education—Textile Institute course in Dyeing and Chemistry; Extension work at Brown University; Executive Training with Alexander Hamilton Institute of New York, N. Y.; working knowledge of one foreign language.

Experience—Dyeing executive on all types of textiles including stock, yarns in the skein, warp and package, and piece goods. Thorough knowledge of all classes of dyes and dyestuffs and their successful practical application in the plant. Extensive knowledge of all types and makes of equipment for dyeing, bleaching, mercerizing and finishing. Laboratory practice in testing, standardizing, purchasing, costing, planning and checking processes, and market analysis. Good manager of personnel for better efficiency in retaining friendly labor relations. Conversant with modern methods for quality production economically secured through better coordination and correct alignment in processing, on cotton, viscose and acetate rayon, silk, wool, worsted, and mixtures. In excellent mental and physical health; excellent references.

A-B-D-1

Education—Special courses in general chemistry.

Experience—Twenty-nine years' experience in application and standardization laboratories of various dyestuff manufacturers. Well versed in the application of dyestuffs to different textile fibers, i.e., wool, cotton, rayon, silk, acetate rayon and unions, using direct, acid, diazo, sulfur, acetate rayon, chrome and after-treated direct dyestuffs. Six months' actual dye house experience on jigs, open boxes, padders, bleaching and mercerizing. Experience demonstrating sulfur and direct colors at various mills. Seeks position as textile colorist in application laboratory and demonstrator.

Age 46; single; references.

A-D-1

Education—Graduate of New Bedford Textile School, 1936.

Experience—Chemist for woolen mill 7 months; technician and salesman for chemical company 1 year and 9 months. Seeks position as salesman or in purchasing department.

Will go anywhere; married; references.

B-5

Education—High School and Textile School Graduate.

Experience—Twelve years overseer of dyeing on directs, vats, naphthols, sulfur, basic and developed colors. Also experienced bleacher. Age 40; married; references.

B-6

Education—Graduate New Bedford Textile School; also post graduate work in advanced microscopy and rayon testing.

Experience—Five weeks in color shop of print works. Wants position in anything associated with textile chemistry, dyeing, finishing and testing.

Single; age 21; references.

B-D-1

Education—General education in England. Chemistry and Dyeing Courses at Huddersfield Technical School.

Experience—Has been a superintendent of dyeing since 1911. Has had experience in worsted mills, hosiery mills and woolen mills. Congenial, sober and reliable. Married; references; age 53.

D-F-1

Education—Graduate of the Massachusetts Institute of Technology, degree of mechanical engineer.

Experience—Four years engineering work consisting of servicing, testing, installing and designing paper box machinery and oil burners. Eight years selling industrial automatic controls, drying equipment and heat treating furnaces. Have made sales market studies and managed a sales office.

Age 36; single, references.

F-1

Education—Two years high school; 6 years evening engineering school; 1 year evening textile school; 1 year industrial management; course in accounting.

Experience—Ten years in an executive capacity in full fashioned hosiery manufacturing business. Familiar with entire manufacturing procedure, finishing dept., dyehouse and office. Specialized in finishing dept. and has good eye for color, finish and quality. Had responsibility of all orders going through mill, stock, grey goods and traffic. Five months in New York sales office. Gets along well with people and contact with employees and customers has always been satisfactory. Previous to hosiery experience was in a mechanical business for 17 years.

43 years of age; single, in good health; will go anywhere.

Unemployment Register

Members of the association who are without employment may obtain from the Secretary, or the American Dyestuff Reporter, application blanks for the Unemployment Register. Personal histories and employment records of applicants may be examined by prospective employers.

Twentieth

ANNUAL MEETING AND CONVENTION

OCTOBER 18TH AND 19TH, 1940

HOTEL COMMODORE

NEW YORK CITY

OCTOBER 20TH, AT N.Y. WORLD'S FAIR

General Chairman

PATRICK J. KENNEDY

Auspices of

NEW YORK SECTION

BERNICE S. BRONNER
Chairman, Ladies Committee

THE chairman of the ladies committee for the twentieth annual meeting and convention will be Bernice S. Bronner. Miss Bronner is thoroughly familiar with the textile industry from the point of view of both the manufacturer and the consumer as she is now with the textile laboratory of Good Housekeeping Institute and was previously with the laboratory of Brown Durrell Co. She is secretary of the American Association of Textile Technologists and is a member of Home Economics Women in Business. She has numerous plans in preparation for the entertainment of the ladies who will attend the meeting, details of which will be announced at a later date.

TRADE NOTES • NEW PRODUCTS

• CIBA RELEASES

The following sample cards have been issued by the Ciba Co., Inc., illustrating new products and processes:

Sample Card No. 1777—Acid and Cloth Fast Colors on Viscose Rayon; Screen Prints with Ciba Intensifier. It is stated that this card illustrates a new process which has not been used previously for the printing of viscose rayon, because a sufficient fixation of the dyestuff has not hitherto been possible. It is said to be now evident that these dyestuffs can be utilized for rayon printing, (with the exception of acetate rayon), if the print paste contains 180-200 parts of Ciba Intensifier per 1000 parts of paste. In this way a much better fixation of the dye is said to take place, with the bright shades of the acid and cloth fast colors being influenced. The prints may also be said to be of good to fairly good fastness to water and washing. In this card only the best fixed dyes are illustrated. After printing, steaming and soaping, there follows an after-treatment with Sapamine KW or Lyofix DE.

Sample Card No. 1781—Cibacete Printing Colors. This card shows water soluble dyestuffs which are said to be well fixed by printing, show good fastness to washing and light and, with the exception of Cibacete Printing Red BD, are free from any tendency to sublime after printing, or discolor the unprinted portions or adjacent pieces.

• GENERAL RELEASES

General Dyestuff Corp., 435 Hudson Street, New York, N. Y., has released circulars describing the following products:

Benzo Viscose Blue R S—a direct dyestuff which produces very bright, royal blue shades on vegetable fibers, said to be distinguished by their good fastness to washing and easy dischargeability. Circular G-180.

Benzo Dark Green B A—a direct dyestuff which is recommended by the manufacturers for dyeing of vegetable fibers and produces full, covered green shades said to be of good fastness to acetic acid, alkali and rubbing and to discharge to a clear white. Uniform shades are also produced on goods containing cotton and wool, or rayon and wool. The color is also recommended for the dyeing of pure or weighted silk. Circular G-178.

• ROHM & HAAS RELEASE

Rohm & Haas Co., Inc., 222 W. Washington Square, Philadelphia, Pa., has recently released the sixth in a series of circulars describing the RHoplex resins. This circular discusses RHoplex W-55, RHoplex W-66 and RHoplex W-77. The series presents a discussion of the outstanding properties of these acqueous dispersions and suggestions for their application to textiles. Copies are available upon request.

• S.O.C.M.A. MEETING

The regular monthly luncheon meeting of the Synthetic Organic Chemical Manufacturers Association was held at the Chemists' Club, New York City, on Friday, May 17th, 1940. The secretary reported on matters of current interest and called attention to the annual outing at Skytop.

• GAEDE DYEING CO.

The Gaede Dyeing Co., Inc., of Paterson, N. J., has been formed with the following officers: Bert B. Allen, president; William Allen, vice-president; Garret Greendyke, treasurer. It is stated that they have taken over the plant and equipment of the Gaede Silk Dyeing Co. and that business is being continued without interruption.

• BOONTON ON OCEAN?

From Philadelphia, Pa., we received a letter this week containing an advertisement, written thereon was: "When did they move the Ocean to Boonton?" But the big thing about this . . . Boonton again gets some advertising and this time through an advertisement in the April 1st issue of American Dyestuff Reporter of Naphthole, Inc., of Boonton, an affiliate of the Wecoline Products, Inc. On the advertisement the address (the largest type) for the company is Boonton and in addition the illustration has "Boonton, N. J." in plain black type—no mistaking it, the plant is in Boonton, N. J. The product advertised is Soluble Luxolene, "an all-American product . . . a better finishing oil with modern improvements. It is very soluble, non-foaming, free from objectionable odors, blends well with other finishing aids," etc., etc.

The reason for our friend's question is that the illustration ("Those Smart Americans Improved on Our Oils"), shows two sides of the ocean, with Boonton on the opposite shore. Well, we're not on the ocean, that's true, but it's just "there" and "here," my friend; figurative, as it were. —"Time Topics" in Boonton, N. J., *Times-Bulletin.*

• ALL GRADUATES PLACED

All textile engineering graduates at Alabama Polytechnic Institute had jobs waiting for them when they received their degrees at the 68th Commencement Exercises.

Each of the 13 graduates had already accepted jobs with Alabama, Georgia, South Carolina, and New York firms before they received their bachelor of science degrees in textile engineering from Pres. L. N. Duncan.

Following are names of the 1940 textile graduates, their hometowns, the phase of textile engineering in which they specialized at Auburn, and the companies by which they have been employed:

Paul Otey Anderson, Uniontown, textile chemistry, dyestuff laboratory of the Ciba Company, New York City; Harry Lleonard Bush, Auburn, textile administration, Avondale Mills, Sylacauga; G. W. Cantrell, Lanett, textile design, colored goods department of the West Point Manufacturing Company, Lanett; T. W. Chiles, Albertville, textile administration, textile sales course of the Callaway Mills, LaGrange, Ga.; R. E. Heathcote, Great Neck, N. Y., textile administration, selling agent for chain of hosiery mills, Heathcote and Company, New York City.

E. P. Hulsey, Birmingham, textile manufacturing, rayon and spun rayon goods department of the Judson Mills, Greenville, S. C.; T. J. Jackson, Auburn, textile manufacturing, tire cord plant of the U. S. Rubber Products Co., Hogansville, Ga.; H. L. King, Jr., Huntsville, textile chemistry, dyestuff laboratory of the Ciba Co., New York City; W. C. Knight, Andalusia, Swift Manufacturing Co., Columbus, Ga.

E. J. Lawrence, West Blocton, textile design, sales engineer for textile manufacturing equipment with the Medley Manufacturing Co.; J. K. Orr, Jr., Atlanta, Ga., textile admiinstration, cellulose acetate manufacturing department of the Celanese Corp. of America, Cumberland, Md.; P. G. Pease, Columbus, Ga.; textile administration, sales training course of the Callaway Mills, LaGrange, Ga.; H. H. Prickett, Auburn, textile administration, sales training course of the Birmingham Paper Company, Birmingham.

• TEXAS GRADUATION

Texas Technological College, Textile Engineering Department, Lubbock, Texas, graduated four students, June 3rd. All received the degree of Bachelor of Science in Textile Engineering.

Ersel Matthews, of Floydada, Texas, will enter the employ of Dunean Mills, Greenville, South Carolina; Robert Altman, of Lubbock, and Neil Stewart, Dallas, Texas, will go to Lanett Mills, Lanett,

Alabama; Lloyd Jackson, Lubbock, will go to Ciba Company, New York. A total of twelve calls for men to fill jobs was received by the school.

Lloyd Jackson received the N.A.C.M. medal. Ralph Budd, president, Burlington Lines, was principal speaker at the Commencement.

• NEW YORK CLUB, PHILADELPHIA TEXTILE SCHOOL

A large and enthusiastic group attended the second meeting of the recently formed New York Club of Philadelphia Textile School, held May 15, at Hotel Collingwood, 45 W. 35th St., New York City. The meeting began with a dinner, and following this the business meeting was opened, under the chairmanship of president Milton Schmidt.

It was noted that membership in the Association has just about doubled itself during the last six weeks, there now being 75 enrolled. Another group of about 100 men, while unable to attend the meeting, have written in, expressing interest in the work and desire to participate in future activities. The secretary pointed out that it had been extremely difficult to compile a complete and accurate mailing list; it is suggested that anyone in the trade who was not notified of this meeting get in touch with any of the officers, either by letter or phone, so as to be sure of receiving future notices.

Pro tem officers selected by the small group at the Association's first meeting were confirmed by the larger group, all being re-elected for a full year. They are: president, Milton Schmidt, of E. F. Timme & Son; vice-president, Dr. George E. Linton, of Central Needle Trades High School; treasurer, Benjamin M. Prager, of L. Bachmann Co., and secretary, Howard P. Galloway, of *Textile World*. The president also appointed various committee chairmen. The Education committee will be headed by Dr. George E. Linton, and will study and recommend any desirable changes in the curricula of the school, and in its physical equipment, machinery, etc., as well as confer with the new dean of the school to obtain his ideas on these subjects. The Finance committee, headed by J. Elmer Hahn, of Lebanon Woolen Mills, will consider matters pertaining to financial cooperation between the Association and the School, and also the subject of scholarship funds, prizes to students for outstanding work, and similar matters. The Publicity committee, which has as co-chairmen Stephen S. Marks, of *Fairchild Publications*, and Howard P. Galloway, will have charge of publicizing the Association, obtaining speakers for meetings, notifying members of forthcoming activities, and other duties of this nature. Other committees are to be appointed as their need becomes evident, and the various chairmen are to fill out the personnel of their groups after study of membership lists.

A resolution was passed, instructing the president to draft a letter to Dr. E. W. France, retiring director of Philadelphia Textile School, expressing the appreciation by the club of his long years of excellent service in the field of textile education. It was also decided that Mr. Earle Heard, incoming dean of the School, be invited to meet with the group at a special meeting to be held the latter part of June, at the same hotel, and that an expression of the Association's wishes for success in his new position be tendered him.

• PRELIMINARY REPORT, SYNTHETIC ORGANIC CHEMICALS, 1939

The United States Tariff Commission issued today a preliminary report on United States production and sales in 1939 of synthetic organic chemicals. The report includes statistics of production and sales of coal-tar intermediates, coal-tar dyes, other finished coal-tar chemicals, and of synthetic non-coal-tar organic chemicals.

The report indicates that 1939 sales of all synthetic organic chemicals valued at $381,917,000 were more than in any preceding year, and exceeded by 38 per cent sales in 1938, a year of poor chemical sales. The increase in coal-tar chemicals was 41 per cent and in non-coal-tar synthetic organic chemicals 36 per cent. Factors that contributed to the peak activity in 1939 are improved business conditions, building up of inventories by both producers and consumers, and increased exports particularly in the last quarter.

Production figures are considerably larger than those of sales since a large part of the total output of synthetic organic chemicals is consumed in further processing by the producing companies. The 1939 increases over 1938 in production for the more important groups were: Intermediates 51 per cent, dyes 47 per cent, coal-tar medicinals 25 per cent, coal-tar resins 68 per cent, and all non-coal-tar synthetic organic chemicals 24 per cent.

Copies of the report may be obtained from the United States Tariff Commission, Washington, D. C., or from the Commission's office in the Custom House, New York City.

• SODIUM SULFATE PLANT

Construction of a large plant for production of anhydrous sodium sulfate, at Dale Lake, Calif., is now being rushed to completion by Desert Chemical Company. An analysis of markets for this product is being made, which will determine the extent of 1941 production. Contemplated for 1941 is 50,000 tons, but this can be increased materially should conditions warrant. Approximately five thousand tons will be available for sample shipments by the middle of July this year.

Methods for the production of sodium sulfate at Desert Chemical Company's properties has been under test for several years, and have been successfully proven on a commercial basis. The company is said to be amply financed for large-scale production, with the board of directors representing important Pacific Coast business interests.

Executive offices of the company are at 4031 Goodwin Ave., Los Angeles, Calif.

• CALCO SHADE CARD

A new and unusually complete shade card for dyeing of woolen and worsted piece goods is announced by Calco Chemical Division, American Cyanamid Company, Bound Brook, New Jersey.

This new guide is very useful both because of the convenience of its arrangement and in the scope of information covered. Actual dyeings on worsted of some 180 acid shades are shown, each containing floaters of cotton, acetate and viscose rayon and silk. For each individual color or shade there is a table of data covering 56 different properties of fastness, and other application details.

Each item in these extensive tables represents current testing by A.A.T.C.C. and other approved methods in Calco's own laboratories. In all, more than 5,000 individual tests were required to prepare this information.

To complete the usefulness of the card, a special ten-page section is devoted to details of the latest application methods, problems of faulty dyeing, and suggestions as to their cure.

As quantities are necessarily limited for such an elaborate guide as this, Calco advises that this new Acid Shade Card can be made available only to those applying through one of the regular Calco representatives.

• FILASEAL R4

Amalgamated Chemical Corp., Auburn St. and Trenton Ave., Philadelphia, Pa., announce production of Filaseal R4, a new resin water repellent finish for hosiery which is said to impart an even film of a durable and elastic resin to the silk fiber. Due to the construction of the resin emulsion, the finely divided colloidal dispersion is said to be uniformly distributed on the hosiery. The finish is claimed to: give an effect of "higher twist," give a clear (but not shiny) effect to the stocking and give a feeling of increased tensile strength to the hosiery when the wearer dons them.

It is stated that the product is easily applied but should not be boiled when dissolving, water at 140° F. very effectively diluting the emulsion which comes in the form of a white paste. Dulling agents, hosiery fillers and softeners may be used

with the product to obtain desirable effects.

It is claimed that hosiery finished with Filascal R4 shows some improvement in the number of cycles run on the Frazier machine and a marked improvement was noticed in the resistance to "light snagging" on the snag testing device sponsored by the research department of the National Association of Hosiery Manufacturers. This latter instrument is used to determine the effectiveness of hosiery finishes, particularly the degree of resistance to snags, pulls and runs. The company further claims that the resin finished stockings show superiority in actual wear tests as regards the number of hours of wearing time.

• RIGGS & LOMBARD, INC., DRY CLEANER

Riggs & Lombard, Inc., of Lowell, Mass. announce that they have obtained exclusive rights to manufacture and sell the Derby Continuous Dry Cleaner.

This machine was invented by Roland E. Derby, chief chemist of M. T. Stevens & Sons Company and was first described on March 10, 1939 to the Northern New England section of the American Association of Textile Chemists & Colorists. It processes wool and worsted and mohair plush fabrics direct from the loom and utilizes a non-inflammable solvent, Dercosol, instead of water, soap and detergents.

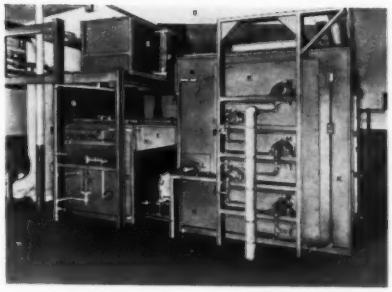
The advantages claimed for this process include:—complete removal of tar, paint, asphalt, creosote and dust spots; tremendous savings in oil due to the fact that it permits the use of the cheapest mineral oil in the picker room; the elimination of stream contamination from oil residue; increase in burling and mending production of 300 per cent or 400 per cent; the elimination of scouring in many instances; the elimination or great reduction in use of soap; a big saving in hot water; a great reduction in redyes and seconds; the creation of conditions favorable to acid fulling; and delivery of fabrics to finishing room with negligible oil content.

It is stated that 96 per cent to 98 per cent of the solvent used in this machine is recovered so that the original high cost of the solvent becomes a negligible factor.

Several of these dry cleaning machines have been in operation in various mills for many months so that the manufacturers have had an opportunity to observe and correct weaknesses in design. Riggs & Lombard state that they are now accepting orders for the latest perfected machine.

• A.I.C. MEDAL

Dr. Gustav Egloff, director of research, Universal Oil Products Company, Chicago, was awared the medal of The American Institute of Chemists for "noteworthy and outstanding service to the science of chemistry and the profession of chemist" at a banquet during the annual meeting of the Institute on Saturday, May 18, 1940, at the Claridge Hotel, Atlantic City, N. J. The presentation was made by Dr. Robert J. Moore, president of the Institute. Colonel George A. Burrell, president of the Atlantic States Gas Company, spoke on the life and accomplishments of the medalist.



Derby Continuous Dry Cleaner

During the afternoon meeting, Dr. Robert P. Fischelis, secretary and chief chemist of the New Jersey State Board of Pharmacy and member of the New Jersey State Board of Health, spoke on "The Status of the Chemist Under the New Food, Drug and Cosmetic Laws."

New officers of The American Institute of Chemists elected for the season 1940-1942 are: president, Dr. Harry L. Fisher, Research Laboratory, U. S. Industrial Alcohol Company, Stamford, Conn.; vice-president, Dr. William T. Read, dean, School of Chemistry, Rutgers University, New Brunswick, N. J.; secretary, Howard S. Neiman, patent attorney, New York; treasurer, Walter J. Murphy, editor, *Chemical Industries*, New York. Councilors, Frank G. Breyer of the consulting firm of Singmaster and Breyer, New York; Dr. Edward R. Allen, technical director, Krebs Pigments Department, E. I. du Pont de Nemours and Company, Newark, N. J.; and Dr. Charles N. Frey, director of the Fleischmann Laboratories of Standard Brands, Inc., New York.

• RAYON MOTION PICTURE

A new educational motion picture with sound, on the subject of rayon, called "Fashion's Favorite," has been announced by the Rayon Division of E. I. du Pont de Nemours and Company.

Planned primarily for training classes of department stores, home economics groups, and women's clubs, the picture is designed to answer common queries about rayon and to clarify misconceptions.

The various steps in the manufacture of rayon yarn are filmed, with emphasis in the narrative on the characteristics of rayon and what these characteristics means in terms of consumer interest.

Prints of the film in 16mm. and 35mm. sizes are available. Requests should be addressed to the Advertising Department, Rayon Division, E. I. du Pont de Nemours and Company, 350 Fifth Avenue, New York City.

• JOINS QUAKER

George H. Rhodes, of Rutherford, New Jersey, has joined the Research Staff of Quaker Chemical Products Corp. of Conshohocken, Pa.

Mr. Rhodes is a graduate of Brown University, Providence, R. I. in Chemistry, Ph.B., later taking two years post-graduate work at the Massachusetts Institute of Technology principally in organic chemistry and organic research.

Mr. Rhodes has been employed at some of the most outstanding mills in the country and will confine his work to the development of resin and organic finishes as applied to textile fabrics.

• NEW STYLE COLOR ANNOUNCEMENT

A new type of dye color announcement has been inaugurated by the Calco Chemical Division, American Cyanamid Company, with the distribution of a bulletin describing Calcomine Brilliant Rubine B prepared by Calco's Technical Service Department. Each of these bulletins will feature a new or important dye that its makers believe deserves special attention. The bulletins are not intended to supplant Calco's regular shade cards, but will supplement them, giving more detailed information about certain selected dyes.

The Color Announcements will be flat and letter size to fit conveniently in a standard file cabinet. They will be tabbed on the side with the color and type of dye for quick reference. The technical staff, in preparing each announcement, has attempted to present a more complete description of recommended practice for use with the particular dye featured. It is this information that, it is hoped, will prove of unusual value to dyers. For some time, efforts have been concentrated to make Calco shade cards of greater usefulness, and this new series of bulletins is one of several steps along this line.

• TRITON 720

Rohm & Haas Co. Inc., 222 W. Washington Square, Philadelphia, Pa., has recently released a booklet entitled "Triton 720" which represents a study of detergent activity with special reference to Triton 720, a synthetic detergent. It is claimed that the product, which is an aqueous paste of a sulfonated ether salt, will accomplish results not ordinarily possible. Copies are available upon request.

Wool Felting—

(Concluded from page 294)

85 ..... 2.15
98 ..... —

Although there is this indication in Schofield's own data of a maximum felting at about 45° C. in agreement with the work of Speakman, Stott and Chang, the remainder of his work indicates some difference between the effect of temperature on the making of a hard felt and the fulling of cloth.

E. OTHER PROPERTIES OF THE FELTING SOLUTION

1. *pH.*—pH has an important effect on the rate of felting of wool. Speakman, Stott and Chang[21] have shown that a minimum felting action is obtained in the pH range 4-8. On the acid side shrinkage increased continually as the pH decreased. This held true down to a pH of 0.5, which was the lowest pH in the experiment. Shrinkage also increased with increasing pH above pH 8 up to a pH of 10.30, obtained with sodium carbonate. This pH was the highest covered in the experiment. Shrinkage with a soap solution of pH 9.92 was higher than in the case of the sodium carbonate solution of pH 10.30, presumably because of the greater lubricating power of the soap solution as contrasted to that of the carbonate solution. It was pointed out that the increased shrinkage at high and low pH coincided with increased swelling at the same points. Further work showed that this coincidence of increased shrinking with higher or lower pH and higher or lower swelling did not necessarily lead to the conclusion that increased swelling resulted in increased shrinking. Thus, if salt was added to the solution, the swelling was decreased while the shrinkage was increased.

2. *Effect of Other Components in the Felting Bath.*—The effect of sodium chloride in increasing shrinkage and decreasing swelling has already been mentioned. It would be of interest to see if any correlation could be worked out between the effects of a lyotropic series on the felting and on the swelling of wool.

F. OTHER FACTORS

1. *Time of Felting*—Schofield[18] gives the following formulae as representative of the effect of time on the felting phenomena:

Felt Making: Volume (V) and time (t)

$$V = 1236 - 695e^{-5.55/t}.$$

Milling: Cloth thickness (T) and time (t)

$$T = a(1\text{-}be^{-kt}).$$

a, b and k are constants.

Cloth tenacities (tensile tests) (S) and time of milling (t)

$$S = 243e^{0.0025t}.$$

Weights of standard area (W) and time of milling (t)

$$W = 0.5 + e^{-81/t}.$$

These are compared to formulae for compression of wool.

(a) Worsted top: Load (L) and contraction (C)—Linear at start, later $L = 150e^{0.22C}$.

(b) Teased heap: Load (L) and contraction (C)—Linear at start, later $L = 3.89 \times 10^5 e^{-c}$.

(c) Baling test (commercial); Pressure (P) and density (d)—$P = 1.3e^{0.07d}$.

Note the similarity of form of these equations.

2. *Mechanical Factors*—Pressure exerted against the material during the felting operation, work done on unit mass of the wool and the characteristics of the motion impressed on the fiber mass each have an effect on the extent to which the wool is felted in a given time. Unfortunately little or nothing is written about these factors.

CONCLUSION

Examining the above rather restricted outline of factors influencing wool felting and the work done so far on these subjects, the authors decided that the following subjects could be profitably studied.

1. The use of felting aids.
2. Study of chlorine and its derivatives to see if it is possible to both increase and decrease the feltability of wool.
3. Study of the effect of alkalies on felting.
4. Study of the effect of a lyotropic series of ions in the felting medium on swelling and on felting.
5. Study of wetting agents with respect to wetting and felting characteristics.
6. Study of the effect of carbonizing on the feltability of wool.
7. Study of the effect of reducing agents such as bisulfites, hydrosulfites, formaldehyde, etc., on the feltability of wool.

Work is now in progress along some of these lines.

ACKNOWLEDGMENT

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Two-Stage Spinning for the Production of

Special Viscose Rayon Filaments

A. J. HALL, B.Sc., F.I.C., F.T.I.

IN the early days of viscose rayon production quite a large amount of research was devoted towards the discovery of the most satisfactory coagulating bath, for it was found that the properties of the resulting filaments depended largely on the manner in which the streams of viscose issuing from the spinnerets were decomposed and converted into solid cellulose. A large number of patented formulae comprising various acids, inorganic salts and protective colloids are available.

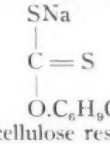
In devising a coagulating bath due regard must be paid to a number of features and among those of prime importance are the following: The cellulose xanthate must be rapidly decomposed by acid (almost universally sulfuric acid is employed) with formation of cellulose, sodium sulfate, and various sulfur compounds among which is found hydrogen sulfide. Connected with this it is important that the cellulose formed has sufficient tenacity to withstand the tension (stretching) on the rayon filaments; the presence of sufficient sodium sulfate in the coagulating bath insures this. Also, the acid must not be allowed to harm the freshly formed cellulose; the presence of protective colloids such as glucose is useful for this. The sulfuretted hydrogen gas should not bubble out of the cellulose filaments into the coagulating liquor since this would cause a degree of disintegration; the composition of the bath should favor a steady non-bubble dissolution of the gas into it. Finally, the salts carried forward in the fully formed rayon should not readily crystallize and harm it.

Connected with the influence of the coagulating bath on the rayon properties is the nature of the stretching to which the filaments are subject on their way through the coagulating liquor and to the point at which they are wound on bobbins or collected in the centrifugal pot. A high degree of stretching reduces the dye affinity of the rayon but at the same time confers on it an increased luster and tensile strength.

All the above features of spinning have in the past received attention from the viewpoint of the production of continuous filament rayon, that is, rayon yarn consisting of long fibers similar to those of real silk in contrast to short fibers such as those of cotton and wool. But during the past five years increasing attention has been given to the manufacture and utilization of cut-up rayon (rayon staple fiber) whose fibers are produced comparable in length with those of the older fibers (cotton and wool) which they are intended to replace or partially substitute in mixture materials. In these developments the conditions of rayon spinning have had to be reviewed and some interesting modifications of the coagulating bath and methods of converting the viscose into solid cellulose have been and continue to be made. It would appear that it is in such modifications that important rayon developments are possible.

In the production of ordinary rayon it is customary to spin the viscose into a coagulating bath which simultaneously makes the viscose insoluble and decomposes it to form cellulose. It is necessary to distinguish these two processes clearly although if one simply watches the operation of spinning rayon filaments it would seem that only decomposition occurs and that this is the only chemical process that matters. By carrying out these two processes separately and successively under controlled conditions it is possible to produce filaments quite different from those resulting when insolubilization and decomposition occur simultaneously.

To understand these developments it is necessary to recall that viscose as used for spinning rayon is a solution in dilute caustic soda of the sodium salt of cellulose xanthogenate having the following decomposition:



In this formula the cellulose residue, $C_6H_9O_4$, is shown in its simplest form but it is more probably $C_{12}H_{19}O_9$, $C_{18}H_{29}O_{14}$, or $C_{24}H_{39}O_{19}$. When a viscose solution is thrown into water there may be partial precipitation but if a considerable amount of sodium chloride or sulfate is present then the sodium cellulose xanthate is quantitatively precipitated. On the other hand, if the water contains a relatively high concentration of acid, say sulfuric acid, then sodium and sulfur compounds are formed, and, particularly, the cellulose residue is split off in its usual insoluble condition.

These properties of viscose indicate that it is possible to spin viscose into a special coagulating bath containing inorganic salts and substantially free from acid to obtain sodium cellulose xanthogenate filaments which can be later decomposed into cellulose filaments as desired. For-

tunately this is so, for the strength of the non-decomposed filaments is sufficient to allow them to be handled without breakage. As a matter of fact the coagulating bath can contain small amounts of acid since it requires a considerable acid strength to decompose the sodium cellulose xanthogenate into cellulose. For instance, this cellulose product is not decomposed by the organic acid, acetic acid.

It will be realized that in ordinary rayon spinning where insolubilization and decomposition of the sodium cellulose xanthogenate take place simultaneously under stretching at a very high rate of travel of the rayon filaments there is but little chance of modifying the character of the filaments so that they are other than straight and more or less smooth filaments. But by separating the stages this opportunity is afforded; the sodium cellulose xanthogenate filaments are susceptible to treatment by various means so as to modify their properties and physical form before conversion into cellulose.

One important feature of this so-called two-stage production of cellulose filaments is that it allows the shape of the filaments to be *permanently* changed. If the fully formed cellulose filaments are subjected to distorting forces so that they are twisted, flattened, or otherwise changed and in this state are wetted or stretched then they return to their original form—the distortion is only temporary. On the other hand, if the sodium cellulose xanthogenate filaments in a distorted form are decomposed by acid into cellulose then the form in which they are thus set has a fair degree of permanency. This is an important fact.

Now that rayon staple fiber (the cut-up fibers) is widely used for the production of yarns in which the fibers are twisted about each other to secure their adhesion and a satisfactory yarn strength, two defects of viscose rayon have been discovered. Firstly, the fibers have such a smooth surface that they slide over each other easily and their ends protrude so as to give the yarn (or fabric in which the yarn may be used) a hairy surface. Secondly, the fibers are so straight that they do not grip each other with that force which characterizes the twisted-ribbon like cotton fibers or the scale-covered and crimped (wavy) wool fibers. It has therefore become apparent that a definite improvement in cut-up rayon fibers would be to produce them with an irregular (reticulated) surface and with a crimp. By carrying out the two-stage production of viscose rayon outlined above a certain degree of success has been obtained in this direction, and some idea of these developments may be obtained by reference to a few selected patented methods. These changes in the character of the fibers are also sometimes accompanied by special effects as regards their luster and softness of handle.

An early process (E. P. 451,860) for introducing a permanent crimp into cut-up viscose fibers is operated thus. The viscose is first spun into a coagulating bath containing a suitable amount of ammonium sulfate, or organic acid, or a sufficiently diluted mineral acid incapable of decomposing sodium cellulose xanthogenate, so as to produce a band of continuous sodium cellulose xanthogenate filaments. This band or bundle, having a total denier of several thousands, is led through a cutting machine to form a mass of short fibers which are plunged into a violently agitated acid decomposing liquor. Owing to their entangled state as promoted by the surging liquor the fibers are converted into cellulose fibers while in a twisted and distorted form. It is claimed that this rough kind of crimp is very stable and withstands the combing treatment which the fibers pass through when being converted into spun yarns.

In one form of this process, the viscose is spun into a coagulating bath containing 25 to 30 per cent of ammonium sulfate and 5 per cent of sodium sulfate. The decomposing acid liquor contains 8 per cent of sulfuric acid and 12 per cent of sodium sulfate.

In a further modification of this two-stage process (E. P. 450,257) a special form of decomposing liquor is used. The sodium cellulose xanthogenate fibers are produced and cut up in much the same manner as described above, but instead of being decomposed in an acid bath they are immersed in a very hot bath of glycerine, glycol, or sulfonated vegetable oil (castor or olive oil). This peculiar method of high temperature decomposition has the effect of fixing in the fibers a crimp sufficient to make them very similar to wool.

Thus viscose is spun through spinnerettes each having about 800 apertures into a bath at 20° C. containing 30 per cent of ammonium sulfate to produce bundles of sodium cellulose xanthogenate fibers which are afterwards cut-up in the usual manner. The fibers are then entered into a glycerine bath at 120° C. to secure the crimped cellulose fibers; these are subsequently washed with water, soaped, washed, dried, and combed into a sliver for later conversion into yarn.

An alternative method of effecting decomposition of the cut-up sodium cellulose xanthogenate fibers is that of drying them at a high temperature. Such a treatment not only gives them a curl and crimp but it leads to their fibers having a rougher surface which increases their adhesion in yarn.

Instead of dry decomposition this treatment may be effected in a boiling solution of an inorganic salt—the temperature of this will exceed 100° C. Such a process for producing viscose fibers having a very fine crimp and whose surface is pitted irregularly is described in E. P. 482,280. The viscose for spinning by such a method is preferably ripened somewhat beyond the usual degree. For example, viscose containing 8 per cent of cellulose and 8 per cent of alkali and ripened to a salt point of 0.5 is spun into a coagulating bath consisting of 90 per cent of methyl alcohol (or methylated spirits). After cutting, the fibers are then converted into cellulose fibers by immersion in a 30 per cent solution of sodium sulfate at 103° C. The luster of the rayon fibers produced in this manner is very low—a true matt appearance.

It will be realized that in processes of the kind described above it is important that the coagulating bath shall be

sufficiently effective to solidify the sodium cellulose xanthogenate filaments so that they do not stick together. Separation of the cut-up fibers would be absolutely impossible if any slight degree of stickiness was produced on the outside of each fiber. The composition of the coagulating bath thus has to be nicely adjusted so that its effect on the viscose is just short of converting it into cellulose for it is under such conditions that the solidified filaments of sodium cellulose xanthogenate will have a maximum of tenacity to withstand the stretching to which they are necessarily subject in their travel.

It has been found possible to assist in attaining these conditions by modifying to a limited degree the characteristics of the viscose being spun into rayon. When a viscose is allowed to stand at room temperature it gradually changes so as to become more and more viscous until at the end of some days cellulose separates out. Without going into detail about the chemical changes which are responsible for this peculiar behavior it can be readily understood that the older (usually termed *riper,* while the period of standing is often termed *ripening*) is the viscose the more easily can it be coagulated to form a non-sticky filament. This suggests that in the two-stage process it would be better to spin into the coagulating bath a viscose which has been ripened to a degree considerably more than usual.

The degree of ripening can be measured by two empirical methods which are generally employed in viscose rayon production. In the first, the ripeness is indicated by the *salt-point* which is the percentage concentration of sodium chloride solution which is just sufficient to coagulate a drop of the viscose when this is added to it. A freshly prepared viscose has a salt-point of around 12 but on ripening this lowers to 3 to 6 when the viscose is normally ready for spinning. By ripening until the salt-point is below 2 to 3 the viscose is better suited for the production of sodium cellulose xanthogenate instead of cellulose filaments.

In the second method, the degree of ripeness is indicated by the number of c.c. of a 10 per cent ammonium chloride solution which must be added to 20 grams of viscose (further diluted with 30 c.c. of water) until complete coagulation occurs. For a freshly prepared viscose this so-called *ammonium chloride figure* is about 12 but this lowers to around 7 for a viscose ripened sufficiently to spin satisfactorily. Sometimes this *ammonium chloride figure* is referred to as the *Hottenroth number* and this is about 4 for a viscose which has ripened so long that it is about to separate cellulose.

Returning now to the manufacture of crimped viscose rayon cut-up fibers by the two-stage method, attention may be drawn to E. P. 509,572. This patented process has for its object not only the production of crimped fibers but also fibers which are voluminous and therefore the more like wool and able to replace this natural fiber.

It is found that it is beneficial to employ a highly ripened viscose and to spin this in a coagulating bath containing a large amount of salt and a low concentration of mineral acid or the formation of sodium cellulose xanthogenate filaments, but according to E. P. 509,572, the fibers resulting from subsequent decomposition of these filaments are not so wool-like as is desired. Thus a special adjustment of these conditions is disclosed. One feature is that a viscose having a low content of alkali is employed and another is that this viscose is ripened excessively, say to a Hottenroth number of less than 5 (about 1 to 3). The low content in alkali tends to accelerate separation of sodium cellulose xanthogenate from such a viscose and so in forming the sodium cellulose xanthogenate filaments it is only permissible to have in the coagulating bath a small content of mineral acid.

With these ideas as a guiding principle the following is an outline of the procedure adopted to produce highly crimped cellulose cut-up fibers having a special bulky character to make them more wool-like. Firstly, an alkali-cellulose (prepared by impregnating cellulose pulp with caustic soda of mercerizing strength and then expressing the excess liquor) is matured for 72 hours at 20° C. and then worked up into a viscose containing 8 per cent of cellulose and 5 per cent of alkali (this 5 per cent is instead of the more usual 7 per cent). It is ripened so as to have a Hottenroth number of 3, and then spun into a coagulating bath containing 300 grams of sodium sulfate and 10 grams of sulfuric acid per liter and maintained at 45° C. The resulting filaments are stretched a suitable amount by passage over rollers, collected into a cable strand form and cut to the desired fiber length. At this stage the fibers have a twisted form so they are entered into an acid decomposing bath containing 10 grams of sulfuric acid per liter whereby they are converted into fully formed cellulose fibers. It is claimed that apart from the special properties of these fibers already mentioned they show no tendency to stick together and pass through satisfactorily the various later processes necessary to convert them into yarn.

Another process (E. P. 481,513) which also relies on the use of a highly ripened viscose solution further demands the use of a very high viscosity cellulose (as starting-out material) or a more highly concentrated viscose. Thus viscose having a salt point of 0.1 (E. P. 443,971 describes the preparation of such highly ripened viscose) is spun into water (the coagulating bath) to give sodium cellulose xanthogenate filaments which are stretched and then treated (in a twisted state) with a decomposing liquor containing 65 grams of sulfuric acid per liter. It is noted that the stretching is prior to the cutting; the fibers are completely free from stretch while in the acid bath.

It will be noticed that mention is made of stretching in this two-stage production of rayon. Such stretching is of importance since it is found that inferior rayon is obtained if it is omitted altogether. Stretching has the effect of orienting the cellulose (or rather sodium cellulose xanthogenate) micellae in the fibers so that they lie more parallel to the fiber axis; this gives them increased tensile strength and resistance to water. If this stretching is applied to the rayon while in its xanthogenate state then

it is not so necessary to apply further stretching while the rayon is being converted into a cellulose rayon. This is fortunate for if the fibers are quite loose during this conversion it is the more likely that they will be set in a distorted (crimped) and wool-like form. The use of stretching in these processes is described in E. P. 512,471. but in this case the two-stage method is employed rather for the manufacture of high tenacity rayon than wool-like fibers.

In this method, viscose containing 7.7 per cent of cellulose and 6.5 per cent of alkali is spun into a coagulating bath containing 24 per cent of ammonium sulfate and 6 per cent of sodium sulfate. The resulting sodium cellulose xanthogenate filaments are then drawn over tension rods and immediately through an 18 per cent solution of sodium sulfate at 50° C. It is this stretching in a hot salt solution which is especially favorable to high tensile strength. Subsequently the stretched filaments are cut-up and decomposed in a boiling liquor containing 29 per cent of sodium sulfate.

There is a slight modification of the two-stage process which according to E. P. 502,199 results in rayon fibers having a highly reticulated surface much resembling that of wool fibers covered with their epithelial scales. There has been some suggestion that such fibers could felt as wool does but it is extremely unlikely that this can be true. As a matter of fact the same suggestion has been made in respect of cut-up casein fibers. It must be remembered that wool felts mainly because each fiber is covered with scales which overlap each other in an *orderly* manner—the scales all point to the tip end of the fiber and they therefore make it possible for the fiber (in a mass of similar fibers) to travel in one direction more easily than another. In contrast the uneven surface of the rayon fibers is very irregular and could not give the fibers this uni-directional impulse to travel which is essential to felting. There seems to be no possible means for ensuring the formation of a uniform type of roughness on rayon fibers and for this reason it is unlikely that in the near future rayon fibers really capable of substituting wool in all its manifold uses can be produced.

However, the following details of this process for making specially roughened wool-like rayon fibers will be of interest. The general method consists of ripening a viscose (containing 6½ per cent of alkali and 7½ per cent of cellulose) for about seven days so that it has the low salt-point of 1 and then spinning this through spinnerets (each containing 372 holes to give filaments each of 4 denier) into a bath coagulating containing 4 per cent of sulfuric acid, 20 per cent of ammonium sulfate, and 10 per cent of sodium sulfate and maintained at 25° C. The resulting filaments are drawn through this bath with a minimum amount of stretching at the rate of 60 meters per minute and are then cut-up by the usual methods.

It will be noted that the composition of the coagulating bath is sufficient to decompose the sodium cellulose xanthogenate threads slowly and it is arranged that these carry with them out of the bath sufficient of this coagulating liquor to complete the decomposition during a period of about ½ hour which is allowed after cutting. At the end of this period when the filaments consist of regenerated cellulose they are purified in the usual manner by desulfurizing and bleaching. Apparently this slow or delayed method of converting the sodium cellulose xanthogenate filaments into cellulose filaments while they are in a cut-up loose condition is effective in producing the special roughness of surface of each fiber. It is further claimed that the fibers are springy and have a luster closely resembling that of wool.

Finally, brief mention may be made of a process (E. P. 424,229) which has for its object the production of rayon fibers having the softness of wool as well as its crimp. The principle underlying this method is that the harshness of handle introduced into ordinary viscose rayon by the high temperature drying of the wet rayon immediately after its manufacture can be avoided if the moisture which it contains is removed by washing with an organic liquid having a strong affinity for moisture. Such a liquid is ethyl alcohol. It is claimed that by using a suitable counter-current system of washing the rayon can be brought to much the same state of dryness as if it had been dried in a hot chamber by the usual method. But by such special removal of water the rayon is left very soft and warm to handle so that it much resembles wool or silk. It would seem that such a process would be very expensive but perhaps it is capable of modification so as to yield similar results at a lower cost.

Sufficient of these modified rayon manufacturing processes have now been described to indicate that there are considerable possibilities in taking advantage of the plastic nature of sodium cellulose xanthogenate filaments to bring them into forms not possible when the viscose spinning solution is instantly converted into cellulose. So far, it appears that it is the plasticity of the sodium cellulose xanthogenate which has received and attracted most attention. But surely some use might be made of the chemical reactivity of these special fibers. A chemical treatment might be applied to modify them internally apart from the changes of physical form producible by distorting forces. This is a field so far left unexplored.

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